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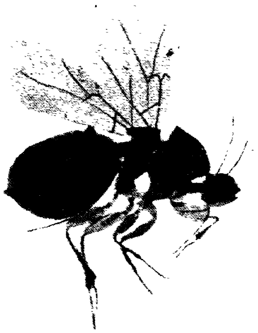

THE GALL WASP

GALL MITES

By the courtesy of Mr. F. J. Ward

MAKERS OF GALLS USED IN THE MANUFACTURE OF INK

Frontispiece

(14621)

PITMAN'S COMMON COMMODITIES
AND INDUSTRIES

INK

BY

C. AINSWORTH MITCHELL

M.A. (OXON), F.I.C.

EDITOR OF "THE ANALYST"; CANTOR LECTURER
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PREFACE

THE aim of this little book is to tell the story of the origin of inks, and to give an outline, as free from technicalities as possible, of the way in which they are made and of the characteristics of good preparations of each kind of ink. It is not intended as a manual for the works or the laboratory, for which purposes my textbook *Inks and Their Manufacture* may be consulted. I have tried, however, to give sufficient details on such points to make the principles of manufacture clear and to interest all who have to deal with inks commercially or to use them in any way.

In writing the book, I have been greatly helped by the kindness shown by several manufacturers in placing information at my disposal and in having photographs specially taken for the purpose. In particular, I wish to thank Messrs. Mabie, Todd & Co., Ltd., for the four excellent photographs showing various stages in the manufacture of " Swan " ink and for many interesting details about their works. I am also grateful to Messrs. B. Winstone & Sons, Ltd., for showing me and giving me full particulars of all the processes used by them in the manufacture of every kind of printing ink, and for the photograph of the special mills used by them in the production of their rotagravure inks.

My best thanks are also due to Mr. James Newman for the specimen of the dried ink sac of a cuttle fish, as used in the preparation of sepia ; to Messrs. Torrance & Co. for the loan of blocks illustrating their machinery for mixing and grinding printing inks; to Mr. T. Roberts for details and illustrations of his filling machines; and to Mr. D. G. Binnington for the description and illustrations of his bottle-washing and cleaning machines.

WHITE COTTAGE, THE COMMON,
AMERSHAM, BUCKINGHAMSHIRE.

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INK

WRITING INKS

I

HISTORICAL OUTLINE

THE antiquity of ink is indicated by the origin of the word, which comes to us through the old French *enque* from the Latin *encaustum,* a name given to the coloured pigment used in baking tiles (whence our " encaustic "). This origin of the word becomes clear when we recall the fact that long before the invention of papyrus the ancient Egyptians wrote with a carbon ink upon earthenware potsherds, which were afterwards baked, so that the writing was made as durable as the material itself.

In all probability the knowledge of the use of such writing ink spread from Egypt into the ancient Greek and Roman world, but the name *encaustum* was, at a later period, applied to the purple ink with which the Roman Emperors signed their names ; whilst the common ink, composed of fine lampblack mixed with a solution of glue or vegetable gum, was known as *atramentum* (from *ater* = black).

Numerous discoveries in Egyptian tombs of ink pots containing the dried-up ink, and of the reed pens by means of which it was applied to papyrus, afford further proof that carbon ink was in common use in Egypt thousands of years before the Christian era.

Ink of a similar kind was also made in China at a remote period; and some years ago an illustrated Chinese MS., dating back to about 2600 B.C., was unearthed, in which a full description is given of the methods then used in the manufacture of the carbon ink which is still known as "Chinese ink." As mentioned later, the details given in this description by Chen-Ki-Souen are essentially the same as those still used in China at the present time.

Carbon inks, with a pigment of lampblack or the like, continue to be used to some extent for writing in Egypt and other countries of the East, and are applied to the paper by means of brushes, reed pens, or steel pens of special construction. In Europe, however, they have long been superseded as writing fluids by iron-gall inks, although they are still used by artists and architects for drawing, and as inks for other special purposes.

Sepia, which is a ready-formed carbonaceous pigment produced by the cuttle fish, is also now mainly used by artists, although during the period of carbon inks it was also occasionally used as a writing fluid. Cicero, for instance, refers to the use of sepia for this purpose.

It is not possible to fix the date at which the ancient carbon inks were generally replaced by the modern iron-gall products, and it is probable that the transition was a very gradual one, the carbon inks being retained by some long after the more convenient iron inks had come into general use.

Making allowance for this innate conservatism, the evidence which can be derived from a study of old documents indicates that iron-gall inks were commonly employed for writing before the seventh century A.D. The subject was studied by Astle, who was Keeper of the Records in the Tower of London; and in 1803 he published a book on the origin of writing, in which he pointed out that the inks in Anglo-Saxon records of the sixth to the tenth centuries were much blacker than those in documents of the sixteenth and seventeenth centuries, and concluded from this fact that the former were carbon inks.

Blagden, however, who also examined the writing on these earlier documents and applied chemical tests to the inks, invariably detected iron, and regarded this as a proof that the writing was in an iron-gall and not a carbon ink. This test, it should be mentioned, was not necessarily conclusive, for it has been shown by Lucas that the carbon inks in many old Egyptian MSS. contain small amounts of iron as an impurity, probably derived from the material used for the preparation of the carbon pigment. At the same time, other inks on documents of the sixth and seventh centuries A.D. have

FIG. 1

ANCIENT EGYPTIAN POTSHERD

Showing writing in carbon ink

been found to contain iron and to show indications of particles of carbon. It is improbable that a mixture of the two types of inks should have been used.

The earliest known reference to the preparation of an iron ink is that given by Theophilus, a monk of the eleventh century, who wrote a cyclopaedia of Christian art, in which he described a writing fluid made from a mixture of an extract of thorn wood with green vitriol (iron sulphate).

The use of green vitriol for this purpose was also described by one of the early alchemists, Albertus Magnus (A.D. 1193–1280), and in 1393 a treatise was published in Paris describing the preparation of ink from galls and green vitriol. The proportions of the ingredients—galls, copperas, and gum—commonly used are given by Peter Canneparius, of Venice, an edition of whose book on inks, written in Latin, was published in London in 1660.

Robert Boyle (1627–1691), too, whose acute mind threw light upon so many things accepted as commonplaces in everyday life, did not overlook the nature of ink, and published the results of his experiments upon the reactions involved in its formation.

In the eighteenth century the question of the correct proportions of galls and copperas to be used was studied by Lewis (1748), by Eisler (1770), and by Ribeaucourt (1792), and varying quantities of these main ingredients were recommended as giving the best results.

But, long before any of these investigations had been published, there was a widespread knowledge of the general principles of making ink. In every large household, recipes for its preparation were to be found in the note-book to which the housewife referred for directions for everything she wanted from the brewing of beer to the mixing of nostrums for every ailment.

Several of these early manuscript books are still extant, and one of them, belonging to the Fairfax family, was published in facsimile some years ago. This contains a number of recipes for making ink, the earliest of which dates back to about the end of the sixteenth century.

The manufacture of ink as an article of commerce does not appear to have developed before the beginning of the seventeenth century, for the earliest reference discovered by the writer is that relating to an ink made by Guyot and sold in Paris in 1609 under the name of *encre de la petite vertu*. It was not until later in that century that an ink factory was established in Dresden ; this preceded by many years the first ink works (Stephens) in this country.

In the earliest types of iron-gall inks, the product was exposed to the air until considerable darkening had taken place in the liquid itself, so that the writing appeared nearly black when the ink was first applied to the paper. In the modern inks this oxidation process is not allowed to take place to any material extent, and writing produced with such inks would at first appear only pale grey on the paper. Hence, to obviate this drawback and to give colour to the writing while the black pigment is forming, a second pigment, such as indigo or an aniline dye, is added as a provisional colour. The use of indigo for this purpose was first described by Eisler in 1770, and was first used in this country by Stephens in 1836. An ink containing a mixture of madder and indigo was patented by the firm of Leonhardi, of Dresden, in 1856 under the name of " alizarine " inks, and the term was retained long after the madder was omitted as superfluous.

The older type of inks is now practically obsolete, and inks containing two pigments, and therefore termed *blue-black*, *violet-black*, and the like, are now in general use. Even in the case of black inks, oxidation is not allowed to take place, but a second pigment is used. The discovery of the aniline dyes was, therefore, of great importance to the manufacturer of ink, for not only were blue pigments made available to take the place of indigo as a provisional colouring matter in black inks, but the soluble pigments which had long been used as coloured inks were also to a large extent replaced by the colours derived from coal tar. In 1861, Cr[illegible] took out a patent for the use of such dyes in ink, and in 1867 a solution of nigrosine was put upon the market under the name of *stylographic ink.*

II

THE RAW MATERIALS FOR IRON-GALL INK

A. TANNIN MATERIALS

THE term " tannin " is used as a group name for a class of substances which are found widely distributed in the vegetable kingdom, and have the characteristic in common of combining with gelatin to form an insoluble compound analogous to leather. They have been roughly divided into two classes, according to whether they form a green compound or a blue compound with iron salts. The tannin derived from hemlock is typical of the former class, and is unsuitable for ink making; whereas the iron-blueing tannins in oak-galls, chestnut bark, and sumach can all be used as the tannin ingredient of ink.

Chestnut Bark. The use of chestnut bark for ink making was patented by Girond in 1825, but is now probably seldom, if ever, used for the purpose, since it has been shown to give ink of a poorer colour than that obtained from other " iron-blueing " tannins.

Sumach, which is the commercial name given to the leaves of *Rhus coriaria* and allied shrubs, is extensively used in dyeing and tanning, and is quite capable of yielding a durable ink. It was shown, however, by Lewis as long ago as 1748 that sumach was inferior to galls for this purpose; and Ribeaucourt pointed out in 1792 that the ink obtained from it had a greenish tint—obviously due to the green colouring matter of the leaves.

Divi-divi is the commercial name of the dried seed pods of a South American shrub, *Caesalpina coriaria,* which was introduced into Europe about 150 years ago. These pods vary in length from about an inch to 3 in. and are chestnut-brown in colour, although frequently they have a mottled appearance due to portions of the epidermis having peeled off. They usually contain from 40 to 50 per cent of tannin, which gives a good bluish-black colouration with iron salts.

Divi-divi is sometimes used in Germany as the tannin material for ink, but has the drawback of containing a somewhat larger proportion of other soluble substances (gums, etc.) than galls.

Myrobalans are the dried fruit of *Terminalia chebula* and allied species growing in India and the East, where they have long been used by the natives for staining leather. They yield an extract which gives a rich blue-black colour with iron salts, and the ink prepared with them is very similar to an iron-gall ink.

Owing to their containing a considerable proportion of non-tannin constituents, myrobalans are not so suitable for writing inks as for copying inks, in which the presence of gums and the like are an advantage rather than otherwise.

In practice, it is usual to roast myrobalans before using them for ink making, the roasting having the effect of giving a darker ink, which clarifies more readily on standing. This process, which is comparable with the roasting of coffee berries, depends largely for its success upon the skill and experience of the workman, for the temperature at which the best results are obtained does not seem to have been ascertained.

Valonia is the name applied in commerce to the acorn cups of *Quercus aegileps* and other species of oaks growing in Greece and Asia Minor.

The cups collected in the spring are richest in tannin. They are left to dry for some time on the ground and

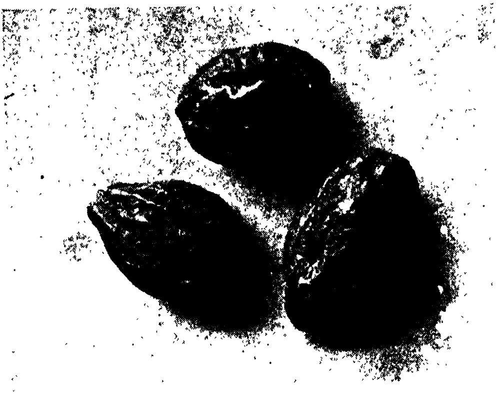

FIG. 2

MYROBALANS

A tannin material used in the manufacture of copying inks

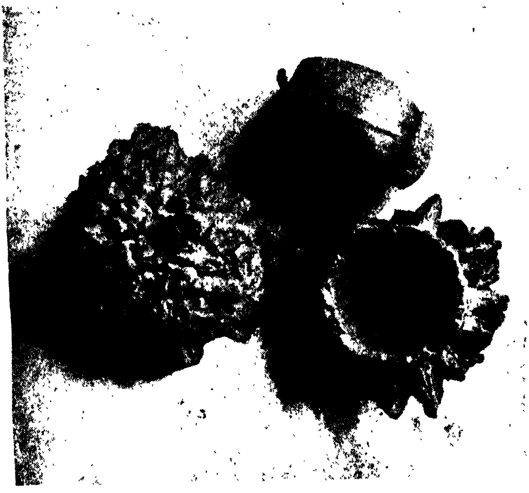

FIG. 3

VALONIA

The commercial name of Acorn Cups used in the manufacture in ink are then sent to Smyrna, where they are stored until a slight fermentation begins and causes the acorns to separate from the cups.

The commercial product, which should consist of only the cups, usually contains from about 32 to 38 per cent of tannin, the proportion varying with the district and the season of collection. The cups are upwards of an inch in diameter, and have rough backs with a structure somewhat recalling that of a pine cone.

Ink made from valonia has a very rich blue-black shade, but is liable to throw down a heavier deposit in the vats than an ink made from galls. If used in admixture with a suitable proportion of galls, however, it makes a good raw material for the manufacture of ink.

Oak Bark contains from about 4 to 10 per cent of tannin, according to the species of oak and the season. It contains not only an iron-greening tannin, but also a compound giving a blue colouration with iron salts. It is, therefore, capable of being used as an ingredient of ink, and has, in fact, been used for that purpose in admixture with galls. It is, however, not nearly so suitable a source of ink tannin as galls, myrobalans, or divi-divi, partly on account of its containing only relatively so little tannin and partly owing to the presence of the iron-greening substance.

Oak Galls. Although all of the foregoing substances have been used, and some (especially myrobalans) are still in common use for the manufacture of ink, the most important source of tannin is the product commercially known as galls.

These are excrescences formed on the branches of various species of oaks by the larvae of certain insects, notably the gall-wasps (*Cynipidae*), the females of which lay their eggs in punctures made into the bark. With the growth of the larva, the gall develops and forms a home for it during this stage of its life-history, until, finally, the larva eats its way out.

The typical example of an English gall is the familiar " oak apple," which in some years develops in great quantities on the oaks in certain districts.

Specimens of English oak apples examined by the writer contained up to 30 per cent of tannin and yielded an excellent ink. Hence, if it were not for the difficulty of collection, these galls might very well be used as a raw material in the manufacture of ink.

Great variations are shown both in the size and form of the galls growing on oaks. Some kinds, such as the Californian " flea seed," are very small ; others, like the ordinary *nut galls* of commerce, have a wrinkled surface ; and others, again, like the French " artichoke galls," form remarkable bulbous excrescences.

Acorn Galls (*Knoppern*), which develop on oaks in the forests of Austria and Czecho-Slovakia, resemble nut galls in appearance ; they are sometimes used in the manufacture of ink. The average amounts of tannin found by Hinrichsen in European varieties were as follows : Morean, about 30 ; Istrian, over 40 ; German and Central European galls, about 15 ; and Knoppern, up to 35 per cent.

Nut Galls, which are also known commercially as **Aleppo, Turkey,** or **Levant Galls,** are produced by a female gall-wasp, *Cynips gallae tinctoriae,* upon a small oak, *Quercus infectoria,* growing abundantly on the Syrian coast and in Palestine.

The larva reaches maturity in about six months, and then eats its way out through a passage which it makes. (*See* Fig. 4.) The empty galls are known in commerce as *white galls* and, as they contain much less tannin than the inhabited galls, are worth considerably less than the latter, which are termed *blue* or *green galls,* according to their colour. The commercial value of a consignment will, therefore, vary according to the proportion of white galls present, the best grades being harvested before the larva escapes.

The galls derived from certain districts are recognized as being of better quality than those from other districts, and therefore fetch a better price. For example, **Smyrna galls** are larger and darker than the Aleppo variety, and often show a larger proportion of white galls; whilst **Tripoli** or **"tarablous" galls are** regarded as of less value than the Syrian products.

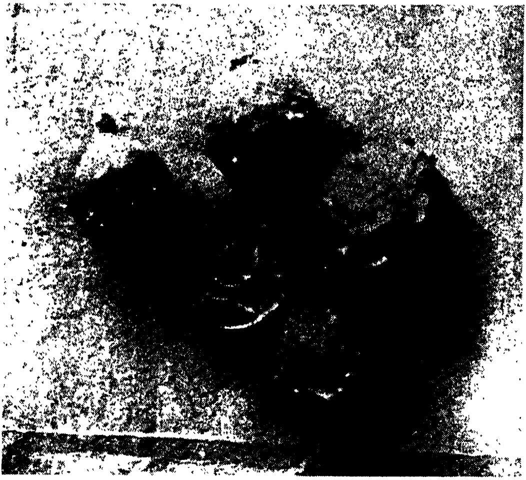

FIG. 4

ALEPPO OR NUT GALLS

The principal raw material for the manufacture of writing inks. A section of a white gall is shown. Also a whole white gall, with the hole through which the larva escaped

Mecca or **Bassorah Galls** are a distinct variety, produced by a different gall-wasp (*C. insana*) ; they contain less tannin than the Aleppo galls, which in the case of good samples may contain over 70 per cent, although frequently the proportion is about 60 per cent or less.

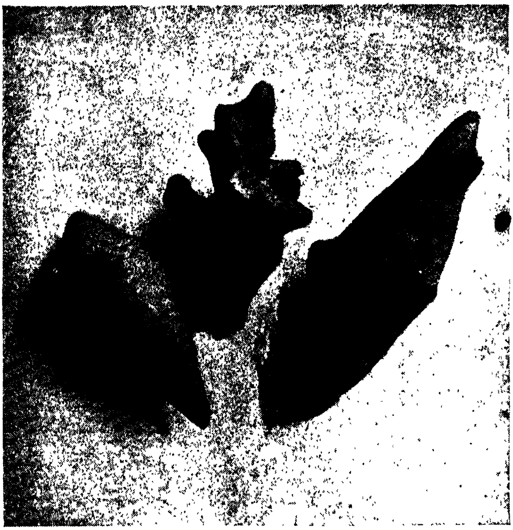

Fig. 5

CHINESE GALLS

An important raw material for the manufacture of writing ink

Chinese and Japanese Galls. These commercial varieties of galls differ materially from nut galls and other kinds of oak galls, both in form and in the fact that they are not produced by gall-wasps on the branches of oak trees, but by small aphides upon the leaf-stalks and shoots of *Rhus semialata* and other species of *Rhus* growing in China, Japan, and Northern India.

In commerce the Chinese and Japanese varieties are frequently sold indiscriminately, although distinct differences have been noted between them, the Japanese galls being smaller and paler in colour than the Chinese, and also differing somewhat in shape.

Chinese galls have long been used for medicinal purposes in China, but did not become known in Europe until the eighteenth century, when they were termed " Indian ears " and regarded as curiosities. It was not until the middle of the last century that they were imported regularly as commercial products. In this country they are used to a much less extent than nut galls as the source of ink tannin, but in Germany have been given the preference over the Aleppo variety.

They have a curious characteristic leaf-like shape, with thin brittle walls of material resembling horn. When broken open, they show a hollow interior, within which is a little dust containing the dried aphides.

It has been estimated that each gall contains about 3,000 of these aphides. Each aphis measures about $\frac{1}{30}$ of an inch in length by about $\frac{1}{40}$ of an inch at the broadest part of the abdomen. The galls are collected by the Chinese peasants shortly before the walls of the gall burst open to allow the larvae to escape, and the aphides are then killed by steaming the galls.

The Japanese galls are collected in the early autumn, immersed in boiling water for half an hour to kill the aphides, dried in the sun for three or four days, and stored in Kiyoto. The aphis is very similar in form to the Chinese variety, but is usually much larger. The difference in the shape of the galls also suggests that the aphides are of different species.

Commercial Japanese galls are usually less horny and have thinner walls than the Chinese product.

The amount of tannin in both varieties of galls is very high, usually ranging from about 65 to 70 per cent. In each case the tannin appears to be of the same type as the tannic acid of nut galls.

An excellent ink can be made with either variety; and, in view of their high proportion of tannin and correspondingly low proportion of inert material, Chinese and Japanese galls must be regarded as exceptionally suitable for the manufacture of ink.

Tannic Acid. The tannin or active principle of gall nuts, when separated in a relatively pure condition, is known as tannic or gallotannic acid, $C_{14}H_{10}O_9$. The commercial product is a glistening powder varying in colour from a pale cream white to brownish yellow, according to its degree of purity. It is fairly soluble in water and dissolves readily in ether or alcohol.

It is frequently used as an ingredient of inks, especially when galls or other materials poor in tannin are being used, and it has the advantage that its use enables the composition of the final ink to be controlled more exactly than is otherwise possible. For this reason, it is used as the principal tannin material of the standard ink with which all German inks are officially compared.

Gallic Acid. When tannic acid is heated with a dilute acid, or is subjected to a fermentation process brought about by certain moulds or yeasts, it takes up water and is converted into a new compound, gallic acid ($C_7H_6O_5 + H_2O$), which crystallizes in glistening white needles. Like its parent, it gives a bluish-black colouration with iron salts, but does not combine with gelatin to form an insoluble compound. It is much less soluble than tannic acid in water, its solubility being less than 1 per cent.

Gallic acid is largely used as an ingredient of inks, either alone or in admixture with tannic acid. Its compounds with iron are stable in solutions free from mineral acid, whereas iron tannates require the presence of a small amount of a strong acid to prevent their depositing. Advantage is taken of this characteristic in the preparation of " acid-free " inks.

B. IRON SALTS

Although gallotannic acid forms coloured compounds with various metallic salts, such as copper, only iron salts have been found suitable for the manufacture of ink. The salt which has long been used for the purpose is ferrous sulphate, but ferric chloride is an ingredient of certain inks, and recently a compound chloro-sulphate of iron has been patented for use in ink making.

Ferrous Sulphate or Copperas. This is the salt which is referred to as *green vitriol* or *copperas* in mediaeval medical treatises, the corresponding sulphates derived from copper and zinc being known as *white vitriol* and *blue vitriol* respectively. The term *copperas*, which is still used in the ink industry, does not, as the word would suggest, imply that it has anything to do with copper. It comes to us through the old French *couperose*, which word was used with the prefixes *verte*, *blanche*, and *bleue* for the three sulphates or vitriols, and probably referred to the method of preparing them by disintegrating (*couper*) the metals with *vitriolic* (i.e. sulphuric) *acid*.

Ferrous sulphate is formed when iron is dissolved in sulphuric acid, hydrogen being meanwhile evolved. The more usual method, however, is to roast iron pyrites, FeS_2, so as to form iron sulphide, FeS, which is then converted into the sulphate by exposure to moist air. The mass is treated with water to extract the resulting sulphate, and the solution is then evaporated in the presence of iron, decanted from the sediment and left to crystallize in pans.

The composition of the salt depends to a considerable extent upon the temperature of crystallization. When formed at ordinary temperature, it contains 7 parts of water of crystallization, $FeSO_4 . 7H_2O$, but if crystallized at 80° C. (176° F.) the crystals will contain only 4 parts of water, $FeSO_2. 4H_2O$. Frequently the crystals have a bright green colour owing to the presence of a trace of ferric sulphate. When exposed to the air the salt gradually becomes yellowish brown on the surface, owing to the formation of a basic ferric sulphate. In this condition the product is termed *rusty*, and ink-makers have found that it is preferable to buy it in that form, probably owing to the fact that the use of such salt promotes the darkening process in the ink.

The normal salt, crystallizing with 7 parts of water, contains 20·08 per cent of iron, and the price at which the commercial product is sold is usually proportional to the percentage of iron. Samples of various commercial consignments examined by the writer have been found to contain quantities of iron varying from 18·77 to 25·43 per cent, the presence of amounts in excess of 20 per cent probably being due to crystallization of the salt at higher temperatures, as mentioned above.

Commercial copperas usually has a slight acid reaction, due to the presence of free sulphuric acid, and this is obviously a point of importance if the salt is to be used in the preparation of acid-free inks.

The double sulphate of iron and ammonium would be free from this drawback of free acidity, and as there would be no difficulty in obtaining supplies of the double salt, it would probably prove a satisfactory substitute for ordinary copperas in the preparation of non-corrosive gallic acid inks.

C. GUM ARABIC

Although gum is not an essential constituent of the ink pigment, which, as has been shown, is a compound of iron and tannin, it has long been used as one of the constituents of a complete ink. The original object of adding gum was to thicken the ink to a sufficient extent to prevent the black particles of colloidal or insoluble iron tannate, formed by exposing the preparation to the air, from subsiding.

In the case of more fluid inks, such as are chiefly made at the present day, the addition of a suitable proportion of gum prevents the ink from dropping from the pen or spreading on the paper.

Gum arabic or gum acacia exudes from the stem and branches of various species of *Acacia* (Nat. Ord. *Leguminosae*). As met with in commerce, it is in the form of colourless or yellowish rounded masses of irregular shape, termed *tears*. These have a glistening surface, but are more or less opaque owing to the presence of minute fissures. The tears often enclose fragments of vegetable débris, which detract from their value. One of the best kinds is sold as **Turkey Picked Gum**, whilst other commercial varieties include **Gedda, Senegal, Australian, Barbary,** and **Talca.**

Gum Senegal, produced by *Acacia verek*, occurs in larger pieces than the Turkey variety; it is of a yellowish red colour, and when mixed with water gives a strong mucilage.

D. COLOURING MATTER

The use of an added colouring matter as a provisional pigment in modern writing ink has already been explained. This is quite distinct from the original use of logwood or indigo as a means of improving the colour of an iron-gall ink.

The indigo used by ink manufacturers is usually bought in the form of a paste, and commercial samples vary very considerably in their purity and, consequently, tinctorial powers.

An aniline blue dyestuff is also almost invariably present in blue-black inks, to give the initial bright blue shade to the writing. The dyes most used for this purpose are **Soluble Blue,** which is the name given to salts of triphenyl-rosaniline-trisulphonic acid and analogous compounds, and **Indigo Carmine** or indigotin sodium disulphonate.

Methylene Blue, which is sometimes used for the purpose, is quite unsuitable, as it readily changes colour and is particularly fugitive.

Appropriate aniline dyes are used for the preparation of *violet black* or *green black inks*, which, however, are much more popular on the Continent than in this country.

E. ACIDS

Iron-gall inks require the addition of a small proportion of a strong acid to render them stable. In the absence of such acid, they will soon be acted upon by the air, and become turbid or throw down deposits in the bottle or in the ink-pot. On the other hand, an excess of acid is objectionable, as it is not only liable to cause the ink to fade, but has also an undue corrosive action upon steel pens.

The acids generally used for the purpose are hydrochloric acid, sulphuric acid, oxalic acid, and, less frequently, tartaric or citric acid. The exact proportion required for each particular mixture is usually based on the results which have been obtained in practice. In some cases the amounts added to English inks are unnecessarily high.

The German standard iron-gall ink, with which other inks are compared, contains 0·25 per cent of hydrochloric acid. This proportion could be materially reduced without affecting the stability of the ink.

In the case of inks prepared from gallic acid (p. 16), no addition of acid is required; but it has not yet been found possible to prepare a standard ink (i.e. one containing more than 0·5 per cent of iron) without the addition of some stronger acid.

F. PRESERVATIVES

Ink, being an organic substance, will offer nutriment to the growth of moulds, which may form pellicles on the surface, or even cause decomposition of the preparation. This has long been known; and in a curious book written by John de Beau Chesne and M. John Baildon in 1573, rhymed directions for making ink are given, in which the following lines occur—

> If yncke ye desire to keep long in store,
> Put bay salt therein, and it will not hoare.[1]

Apart from the fact of its not being a strong antiseptic, there are several objections to adding salt as a preservative to ink.

Another preservative met with in old formulae for inks is vinegar.

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[1] Anglo-Saxon *har* = hoary or grey; in allusion to the greyish-white colour of the mould.

In the modern inks the substances now commonly used for the purpose are carbolic acid or phenol, and, less frequently, salicylic acid or a salicylate. The former is usually considered the more satisfactory by ink makers.

III

THE MANUFACTURE OF IRON-GALL INKS

Proportions of Galls to Iron. The proportion of galls to copperas to be used will depend, firstly, upon the amount of tannin in the particular consignment of galls; and, secondly, upon the extent to which the tannin is extracted.

In the older recipes, the amounts prescribed varied from 3 parts of galls to 1 of copperas (Canneparius, 1660; Lewis, 1748) to 2 parts to 1 (Ribeaucourt, 1792); and, in the other direction, to 4 parts to 1 (Eisler, 1770) and 5 parts to 1 (Reid, 1827).

These proportions were based upon the results of practical experiments with nut galls, which doubtless contained very different amounts of tannin, and not upon the theoretical requirements based upon experiments with pure gallotannic acid.

The writer's experiments have shown that, if allowance is made for these factors, good results are obtained by the use of approximately the old formula of 3 parts of galls to 1 of copperas, but still better results by using 3 parts of pure gallotannic acid in place of 3 parts of galls.

This would correspond with about 5 parts of nut galls, 4 parts of Chinese galls, 8 parts of divi-divi, and 11 parts of myrobalans.

There are, however, various non-tannin substances in galls which, although not contributing directly to the tinctorial properties, yet have an influence upon it. The presence of these other substances (gummy matters, etc.), and the possibility that the gallotannic acid has undergone more or less change in the galls, are sufficient to account for the fact that, in practice, the theoretical amounts of galls do not always give such good results as the old formula of 3 parts of galls to 1 of copperas.

In all cases, therefore, the formula should be based on the proportion of tannin in the galls or other raw material, and modified in accordance with the results which the particular consignment gives in practice. For example, as a general rule, a fifth less of Chinese galls than of Turkey galls (gall nuts) can be used, and this allowance can be made when a mixture of the two kinds of galls is employed.

Infusing the Galls. In order to extract the essential tannin substances, it is necessary to make an infusion of the galls in water, and for this purpose they are first crushed to expose the interior surface, and soaked for some days in the suitable proportion of water. The amount of water will naturally vary with the strength of the final ink, being less for a standard ink than for an ordinary writing ink.

The proportions given in several old formulae are 1 part of galls to 30 parts of water, or white wine ; and Canneparius (1660) quotes an old Italian rhyme giving this ratio.

Powdering the galls is unsatisfactory, for, although it enables more tannin to be extracted from them, it also causes other constituents to separate, with the result that it is afterwards difficult to get the ink clear.

After the tannin has thus been extracted from the crushed galls, a fairly clear yellowish solution is obtained. This is drawn off from the extraction vats and is then ready for the addition of the other ingredients.

In some works the extraction of the tannin is facilitated by mixing the crushed galls with straw (to promote drainage) and putting the mixture in a narrow vat with a perforated false bottom. The greater part of the total quantity of water is warmed and poured over the gall mixture, and gradually percolates through it into the space below the false bottom, from which it is drawn off through a cock connected with a pump, which delivers it again on to the top of the "goods" in the vat. In this way there is a continuous circulation of the extract, which gradually becomes richer in tannin, and finally the extract is drawn off into the mixing vats and replaced by the remaining portion of water, which washes the extracted tannin out of the mass. A higher yield of tannin is thus obtained in a shorter time than by simple infusion of the galls.

Mixing and Maturing the Ink. The extract of tannin, whether obtained by slow infusion of galls or immediately by dissolving the weighed quantity of gallotannic acid, tannin extract, or gallic acid in water, is placed in the mixing vats and stirred up with the weighed quantity of copperas dissolved in a small amount of water.

At first there is but little alteration in the colour, but after a few minutes the liquid becomes faintly bluish, and the colour gradually darkens from the surface downwards, as the air slowly acts upon the iron tannate.

If strong acid were not added, this change would continue until a succession of insoluble deposits of iron tannate was formed, as was the case in the old type of inks.

As has already been explained (p. 19), a small addition of an acid, such as hydrochloric acid, is added to the modern type of inks, to retard this change in the ink in bulk and to leave it to take place, in the main, after the ink has been applied to the paper.

By the courtesy of FIG. 6 *Messrs. Mabie, Todd & Co., Ltd.*

MATURING VATS

View of a number of ink vats in one of the factories of Messrs. Mabie, Todd and Co. These vats each hold upwards of 1,000 gallons

The definite quantities of indigo, aniline dye, or other colouring matter, and gum arabic and preservative, are also added to the ink, which is then left to mature for at least a month.

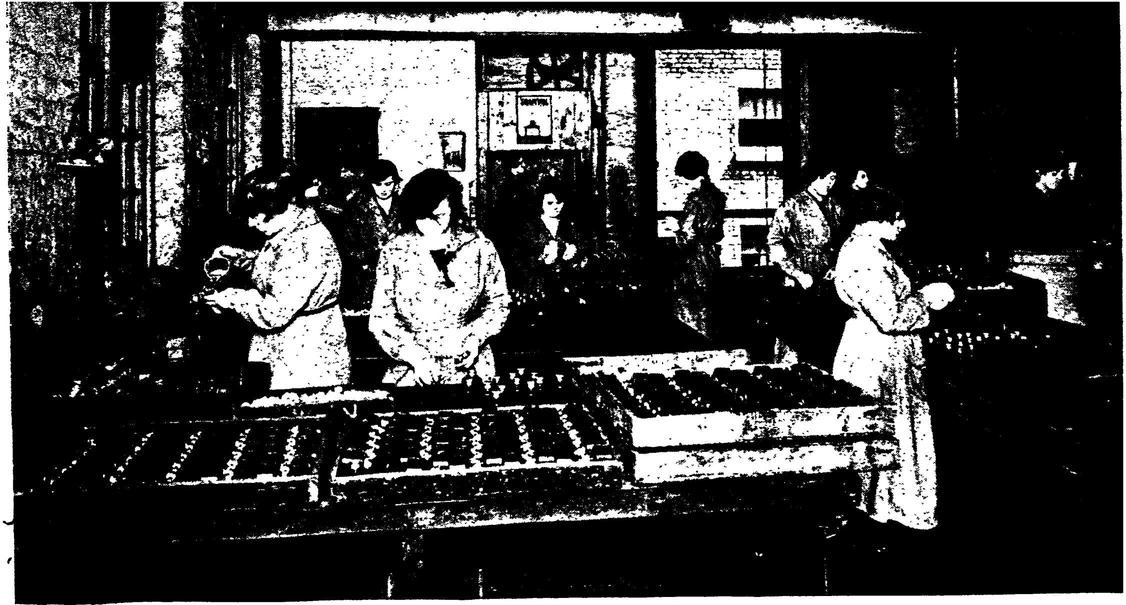
In this maturing process, a large deposit of sludge, composed of compounds of iron with gums and other non-tannin constituents of the galls, is formed, and this carries down with it some of the colouring matter and tannate from the ink. It is essential that this clarification should take place in the vats, since otherwise deposits would be formed in the ink after bottling.

In the case of inks prepared from gallotannic acid, there are fewer impurities present and the clarifying process is complete in a much shorter time, and this applies still more to the so-called "acid-free" inks prepared from gallic acid.

As an illustration of the ingredients of an iron-gall ink, the following formula for the preparation of the German Standard Ink may be quoted: Gallotannic acid, 23·4 parts; gallic acid, 7·7 parts; copperas (ferrous sulphate), 30 parts; gum arabic, 10 parts; hydrochloric acid, 2·5 parts; and carbolic acid, 1 part in 1,000 parts of water. This mixture is allowed to stand for at least four days and then decanted from any deposit. A suitable proportion of a soluble aniline dye of the desired colour is also added.

Bottling the Ink. After the ink has matured and is quite bright, it is put into the bottles, either by hand or by means of a filling machine. The machine has the advantage of speed, but has the drawback that, if it does not work absolutely perfectly, some of the ink is spilled on the outside of the bottles and, owing to its staining power, is not always easy to remove, especially in the case of earthenware bottles.

When the bottles are filled by hand, a sufficient



By the courtesy of FIG. 7 *Messrs. Mabie, Todd & Co., Ltd.*

BOTTLING INK BY HAND ("SWAN" INK WORKS)

quantity of the ink is drawn off to fill a small open wooden vessel, and from this it is dipped by means of dippers made of gutta-percha or some other material not attacked by ink and graduated to remove the exact quantity required to fill the bottle.

Or, the ink may be drawn off in small earthenware jugs, from which it can be poured into the bottles.

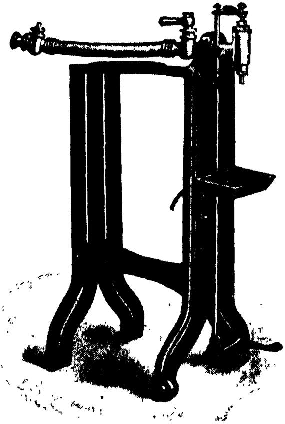


FIG. 8

" NELSON " FILLING MACHINE

Opinion in the industry differs as to the relative advantages of bottling by hand or by machinery. Those who advocate the use of the hand method contend that what is gained in the speed of filling is lost by the extra handling required.

Fig. 8 represents the " Nelson " Filling Machine, made by the Roberts' Patent Filling Machine Co., for use in ink factories and the like. This machine is coupled to an outlet pipe from the ink vat. The vat (not shown) is to the left, and the flow of ink is controlled

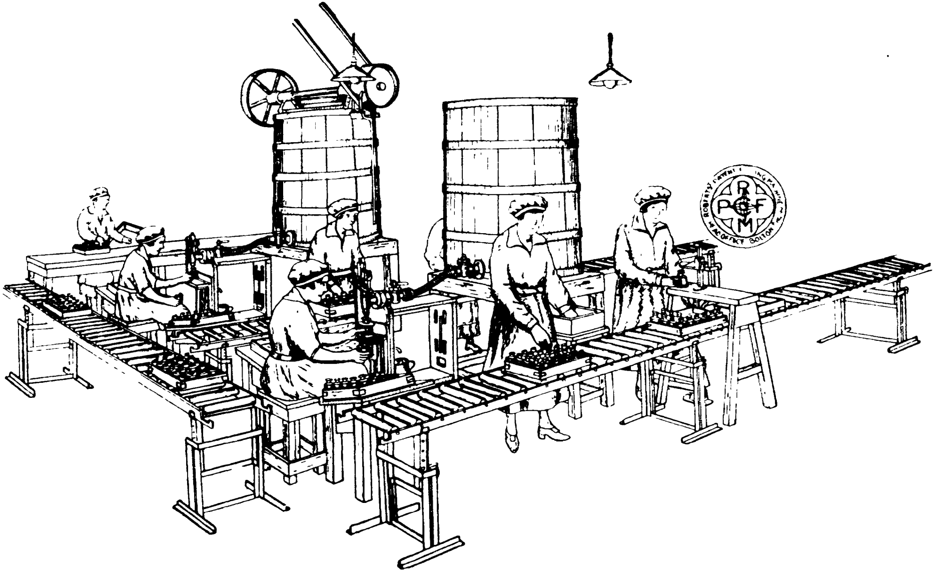


FIG. 9

COMBINATION OF BOTTLE WASHING AND FILLING MACHINES by regulating the vat cock shown on the extreme left of the coupling pipe, or the machine back tap at the other end of this pipe. The bottle to be filled is held in the left hand, and a second bottle is held in the right hand, ready to be placed on the nipple the moment the valve is closed. This valve is controlled by pressure applied by means of the foot to the lever at the base of the machine. The filled bottle is transferred to an inclined tray with the left hand, which then grasps the new bottle beneath the nipple, while a fresh bottle is again picked up with the right hand, and so on.

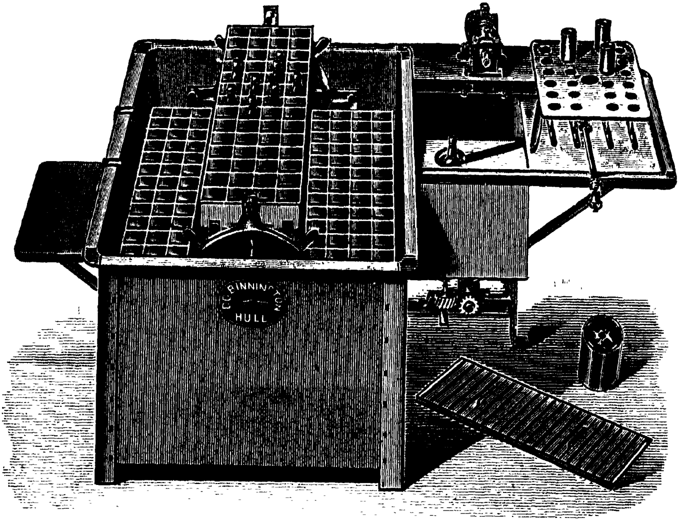
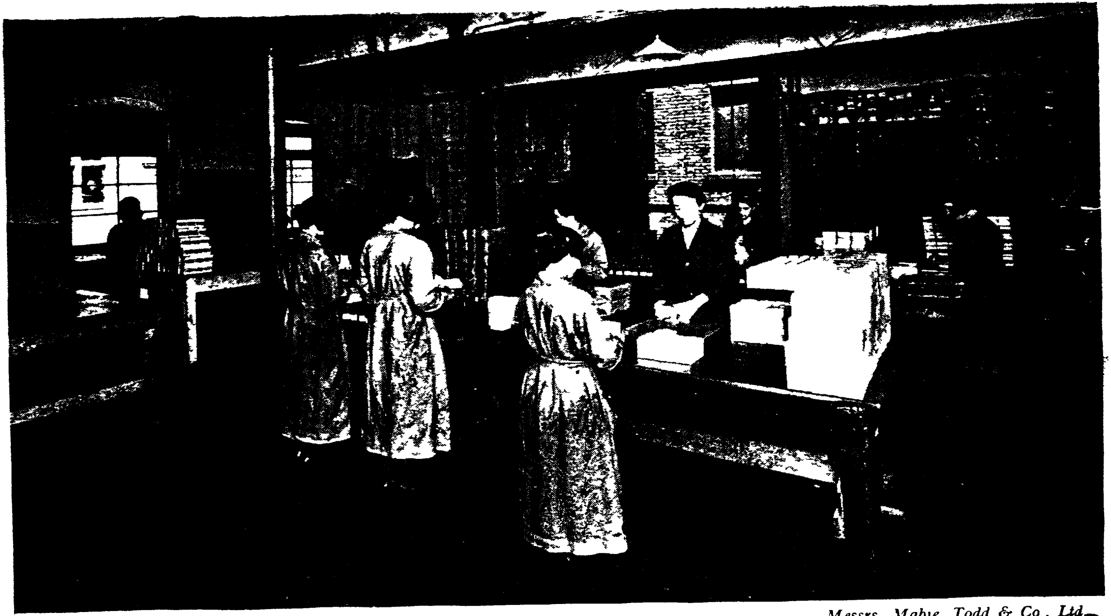


FIG. 10

BOTTLE-WASHING MACHINE

With practice, an expert worker can fill over 2,500 bottles per hour.



By the courtesy of *Messrs Mabie Todd & Co, Ltd.*

FIG 11

LABELLING "SWAN" INK

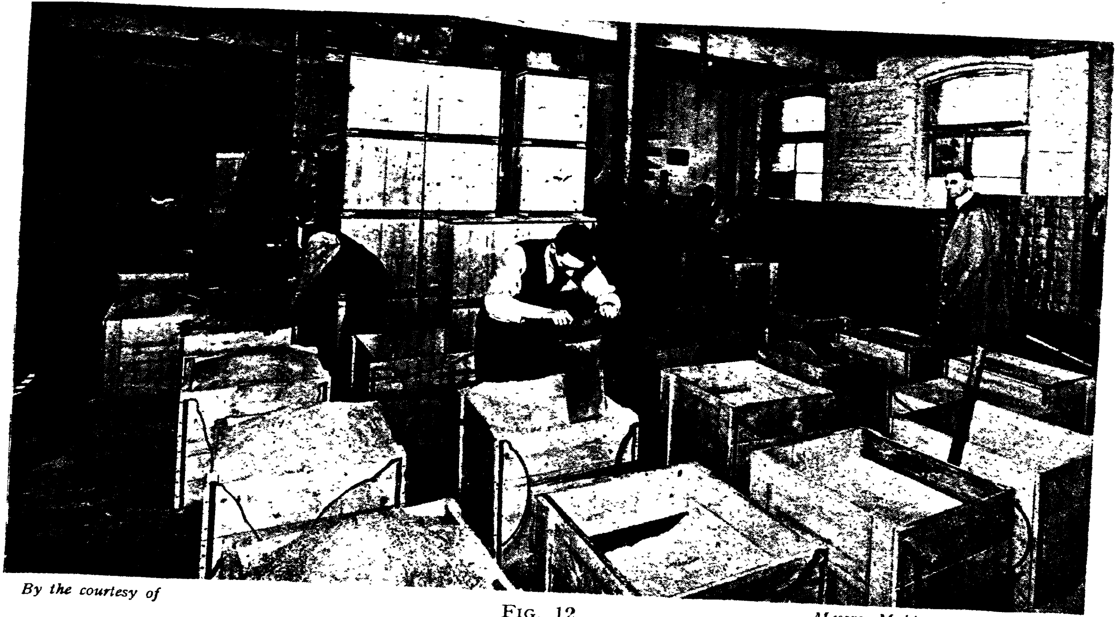
Still greater advantage may be obtained by using a system of automatic delivery to the filling machine by means of conveyor rollers. The bottles are conveyed direct from the washing machine to the filling machine in trays constructed to hold an exact number of bottles, so that the counting is done automatically. The worker at the filling machine takes the bottles from one tray and transfers them, when full, to another. This method of working is shown in the accompanying diagram (Fig. 9), for which the writer is indebted to Mr. T. Roberts.

It is essential that the bottles should be absolutely clean, and free from dust or any particles of straw from the crates in which they were packed. In the larger works, bottle-washing machines, such as those shown in Fig. 13, are used.

This illustration represents one of Binnington's Soaking, Brushing, and Rinsing Machines, which is capable of dealing with eighty to ninety dozen bottles per hour.

The case of dirty bottles is placed on the box rest, shown on the left of the tank, and the bottles are transferred to the uppermost soaking tray in the tank. When this is full, the loose sinker, shown on the ground, is placed over it to prevent the bottles floating when they are submerged in the water. A stop catch at the end of the tray is then withdrawn, and the cross arms which support the soaking trays are made to revolve, until the next soaking tray (to the right) takes up the position, at the top, of the tray just filled. As soon as sufficient water has entered the bottles in the first tray, the sinker can be removed and used to cover the second tray, and so on, until all the trays have been filled.

After thorough soaking, the dirty bottles are removed from the successive trays as they again come to the top,



By the courtesy of *Messrs. Mabie, Todd & Co., Ltd.*

FIG. 12

PACKING BOTTLES OF INK ("SWAN" INK WORKS) and each bottle in turn is placed in the circular opening (shown to the immediate right of the soaking tank), where it is brushed outside for a few seconds by revolving vertical brushes. It is then removed and brushed inside by means of a revolving brush (not shown), which projects forward over the brushing tank. Finally, the bottles are transferred, mouth downwards, to the revolving rinser (extreme right), and each is rinsed inside by means of a jet of clean water from a series of nozzles controlled by a valve beneath the rinsing tray.

The bottles are made either of a somewhat coarse glass or of earthenware. In the past, too little attention has been paid by ink-makers to the composition of the glass bottles ; but within the last two years it has been shown by the writer that the presence of free alkali in the glass will neutralize the acid of the ink, and eventually cause the ink itself to turn brown and throw down a deposit.

Certain makes of glass bottles were found to contain so much free alkali that they gave a strongly alkaline solution after being left in contact with distilled water for a few minutes. It is probable that to this hitherto unsuspected cause have been due many of the previously inexplicable troubles to which all ink manufacturers have occasionally been liable. The remedy, of course, is to test fresh supplies of the bottles and to reject those which show any trace of free alkalinity.

Labelling and Packing. The bottles are corked immediately after filling, and are then wiped clean and a label affixed by hand. The labelling machines used in some industries are not altogether suitable for bottles of ink, as the advantage of the speed at which they work is neutralized by the drawback that a machine requires the attention of two persons. The smaller bottles, containing ink for fountain pens, are put up in cartons, as is shown in the illustration (Fig. 11), which represents the process as carried out in one of the " Swan " ink factories.

These cartons can then be packed direct in wooden boxes made to hold one or two dozen.

Larger bottles, such as the well-known earthenware quart bottles used in offices, are tightly packed in sawdust in wooden boxes, as is shown in the illustration (Fig. 12).

IV

METHODS OF TESTING INK

Standards for Iron-gall Inks. The experience of centuries and the results of scientific experiments, such as those referred to in the preceding pages, have brought cumulative proof that inks made from iron salts and galls, or other tannin-containing materials, are the most generally suitable preparations for writing, especially for documents where permanence is a main essential.

An examination of the MSS. in the British Museum will show that the ink on some of the earliest specimens is of a rich brown colour, which shows no indications of fading. On the other hand, the ink on some of the documents appears very faint and has obviously faded considerably. A notable instance of the kind may be seen in the entry in Milton's Bible of the birth of his daughter Deborah on " the 2nd of May, being Sunday, somewhat before three of the clock in the morning, 1652," the ink of which is very pale in colour.

These variations in the permanence of old writing in iron-gall inks are in all probability due to variations in the proportions of tannin and iron in the inks, for it is essential to good ink that there shall be a sufficient amount of each main ingredient and, in particular, that the iron shall not fall below a definite minimum.

In view of these considerations, the Prussian Government in 1879 prohibited the use of aniline inks for documentary purposes, and made it obligatory to use only iron-gall inks for this purpose.

Nine years later, definite regulations for documentary inks were laid down, and the amounts of iron and tannin substances derived from galls were specified.

Owing to the impossibility of identifying with certainty the origin of the tannin in ink made from materials (e.g. chestnut bark) other than galls, these rules met with much criticism, and in 1912 new regulations were passed in which the source of the tannin in " iron-gall " inks is not specified. For purposes of classification, writing inks are classified in these regulations into: I, Inks for documentary and official purposes; and II, ordinary writing inks; and the latter again are subdivided into (*a*) iron-gall inks, and (*b*) logwood and dyestuff inks.

Documentary Inks are defined as inks made from iron and galls, and giving dark writing after an exposure of eight days to the air. They must contain at least 2·7 per cent of gallotannic and gallic acids (source not specified), and not less than 0·4 per cent or more than 0·6 per cent of iron, and the ratio of the tannin substances to iron must fall within the limits of 4·5 : 1 and 6·75 : 1. The ink must remain stable for at least fourteen days when exposed in an ink-pot, must flow readily from the pen, and must yield writing which is not sticky.

Ordinary Iron-gall Writing Inks must contain at least 1·8 per cent of gallotannic and gallic acids, and not less than 0·26 per cent or more than 0·4 per cent of iron, the ratios between these constituents being the same as in the documentary inks.

Logwood and Dyestuff Inks are not officially tested.

Rules for examining inks are also given in the Prussian regulations, including the chemical methods to be used in estimating the amounts of gallotannic and gallic acids and of iron, and methods for testing the writing.

For example, sheets of standard paper are stretched in a frame inclined at an angle of 45°, and a measured quantity of ink is delivered on to the top of the sheet from a pipette fixed in a definite position in relation to the paper. The ink flowing down the sheet makes a broad stripe, and this is compared with a similar stripe made in the same way and at the same time with a standard ink of specified composition.

The paper with coloured stripes of the two inks is exposed for eight days to the air in diffused daylight, and is then cut horizontally into strips, which are immersed in water, in 50 per cent alcohol, and in 80 per cent alcohol respectively. No perceptible bleaching of the ink should be observed in any of these tests.

These Prussian regulations were adopted many years ago by some of the State Legislatures in the United States, notably Massachusetts, and some of them are embodied in the standard specifications for inks of the United States Bureau of Standards.

Until about three years ago there were no official specifications for writing ink in this country; but inks submitted for Government contracts are now required not only to have certain physical properties, but also to contain specified amounts of certain ingredients. For instance, inks for documentary or record purposes must contain not less than 0·5 per cent of iron, and fountain pen inks not less than 0·2 per cent. Few of the commercial inks on the English market fall below this minimum requirement of 0·2 per cent of iron, and the usual proportion is between 0·25 and 0·3 per cent. Inks containing the quantities specified for record purposes (0·5 per cent) naturally contain also more tannin and gums, and are too thick to be popular for general purposes. In fact, most of the well-known blue-black inks are suitable, so far as regards their consistence, either for fountain pens or as ordinary writing inks.

Total Solid Matter. Provided that the ink contains the specified amounts of iron and tannin acids, the Prussian regulations do not require it to contain any definite proportion of total solids ; and inks which answer to the specifications may, therefore, show considerable variations in their content of gums and other non-tannin substances according to the particular raw material used in their manufacture.

For example, an ink prepared from pure gallotannic and gallic acids would contain much less solid matter than an ink made from galls, divi-divi, or myrobalans, and for many purposes would require the addition of a larger amount of gum arabic.

The standard iron-gall ink used for comparison in the German tests contains, as has been mentioned, 2·34 per cent of gallotannic acid, 0·77 per cent of gallic acid, 3·0 per cent of copperas, 1·0 per cent of gum arabic, 0·25 per cent of hydrochloric acid, and 0·1 per cent of carbolic acid. Making allowance for the water in these ingredients and for a certain amount of deposition in the ink, the final product will contain approximately 5·0 per cent of total solids.

In the case of English commercial inks, this amount of solid matter is usually only found in those preparations which are used as black inks for record purposes, whilst most of the blue-black inks on the market contain from 2·5 to 4·5 per cent. For fountain pen inks, an amount approximating to the lower figure is the more suitable, since it is an essential requirement of such ink that it shall flow readily from the reservoir of the pen and not leave deposits on the pen nib.

The inks made for record purposes by English manufacturers contain from about 5 to 7 per cent of total solids, and one well-known ink contains nearly 8 per cent. In view of other requirements of a good ink, this amount appears excessive. None of these very heavy inks would be suitable for using in fountain pens.

Acidity. The German regulations do not contain any specification as to the permissible amount of acid in an ink. If there is an excessive amount, the stripes of ink made in the test, described in the official methods of examination, will darken too slowly, since acid retards the formation of the pigment of iron tannate.

These regulations, however, do not take into consideration the corrosive action of ink upon the pen nibs, which will vary according to the nature and quantity of the strong acid used. (*See* p. 19.) A practical test of acidity devised by the writer and now adopted, without acknowledgment, by the United States Government in their official specification for ink, is to leave a weighed steel pen nib in a measured quantity of the ink and to weigh it again after a given period. In applying this test to a widely-advertised ink, it was found that the pen had lost 5·18 per cent in weight in a month, and that the ink had become a semi-solid mass.

Although, as has already been pointed out, the presence of a certain proportion of a strong acid is essential, excepting in the case of the so-called " acid-free inks " made from gallic acid, the amount of acidity shown by the above test is unquestionably excessive and could be materially reduced without detriment to the other properties of the ink. Even the proportion of hydrochloric acid (0·25 per cent) in the German standard ink formula, quoted above, is higher than is necessary to ensure stability.

The writer has published elsewhere (*Analyst,* 1921, xlvi, 129) a series of analyses showing the amounts of strong acid present in typical commercial inks, and has devised rapid methods of estimation which are more suitable for the purpose than the somewhat crude pen test mentioned above. In future official specifications as much attention ought to be paid to this important factor as to the other properties of the ink.

Physical Properties of the Ink. A good writing ink should not only produce marks which darken rapidly and remain permanent on exposure to light and air, but should also have certain subsidiary qualifications. Thus, it should flow readily from the pen without dropping as blots, and should penetrate rapidly into the fibres of the paper without spreading too freely over the surface. These qualities are best tested by physical methods, although a chemical examination of the ink will also afford information as to how it will behave.

The comparative rate at which different inks will flow from a pen can be rapidly measured by determining the relative viscosity, that is to say, the rate at which a measured quantity of the ink will flow through a small orifice, as compared with the time required by water to flow through the same opening at the same temperature. For example, in comparative tests, water took 40 secs. to flow through the orifice, whilst the same volume of a very fluid ink took 42 secs., heavy writing inks up to 55 secs., and thick copying inks from 60 to 70 secs.

In the German official methods, the degree of fluidity is judged by the behaviour of the ink in the stripe test (*supra*). At the point where the pipette is brought in contact with the stretched paper, the standard ink forms an oval-shaped head, and the stripe beneath is nearly as wide as the head. This is regarded as the typical form for a documentary ink. In the case of copying inks, the oval head and following stripe are somewhat narrower than the standard, whilst the more fluid inks (of the fountain pen type) produce a stripe with a much wider head than the typical ink stripe ;

whilst the lower part of the stripe contracts to a band much narrower than the head.

The penetration of the ink into the fibres of the paper depends not only on the fluidity, but also on the proportion of acid present. It is a factor which will vary greatly with the character of the paper, and, in particular, with the nature and amount of the sizing. Hence, in applying any tests, it is essential always to use the same standard paper and to compare the results of practical tests made under the same conditions with a standard ink of the same type.

It is commonly accepted by ink manufacturers that inks of the " acid-free " type (Gallic Acid Inks, p. 16) have a lower penetrating power than inks which contain a strong acid. This contention, which does not appear to have been definitely established, could probably be proved or disproved by means of tests in which the degree of penetration was measured with the aid of a micrometer. It seems probable, however, that an acid ink would penetrate more rapidly through sized paper than an ink not containing a mineral acid or strong organic acid.

Keeping Qualities of Ink. An iron-gall ink which has been properly matured will usually keep quite free from sediment for a very long period, if protected from light and air, although sometimes it may form a small sediment if exposed to a great change of temperature. The influence of the alkalinity of the bottle-glass upon the stability of the ink is also a most important factor which must, in future, be taken into consideration whenever the ink in an unopened bottle shows any indication of change. (*See* p. 34.)

The presence of any skin over the ink in an unopened consignment of ink, however, may not be due to any chemical alterations in the ink, but to the growth of mould-fungi or bacteria, and then points to insufficient preservation of the ink.

To ascertain the keeping qualities of an ink, it is necessary to make a practical test by exposing a small sample to the air under standard conditions.

The official method used in examining German inks is to allow the bottle to stand for three days at a temperature of about 60° F., and then to withdraw a measured quantity of the ink from the middle of the bottle by means of a pipette. This is passed through a filter, and one-half of the filtrate is exposed to the air in a wide-mouthed glass cylinder, over which is placed a piece of paper to exclude dust.

Any ink which forms a pellicle on the surface, or begins to throw down a deposit within fourteen days, is not regarded as an ink of the first class, whilst inferior inks will begin to decompose even after a day or two. It was found that when thus tested, under normal conditions of temperature, the standard comparison ink (preserved with 0·1 per cent of carbolic acid) did not show any signs of change for three weeks, after which a skin formed over the surface, and small particles began to separate.

The official requirements for the keeping qualities of inks were based on the results of the test.

V

LOGWOOD AND OTHER BLACK WRITING INKS—COLOURED INKS—COPYING INKS

LOGWOOD INKS

THE dye so well known as logwood is extracted from the wood of *Haematoxylon campechianum*, which grows in Mexico and other Central American countries, and in the West Indies. When first imported into Europe, in the sixteenth century, it was regarded as an inferior dye ; and an Act was passed prohibiting its use in England, and was not repealed until 1661.

Logwood is known in commerce in the form of fine chips, from which the dyestuff can be extracted with water ; but ink manufacturers more frequently purchase it as an extract in the form of a heavy liquid or solid paste.

Haematoxylin, the active principle in the wood, is converted, when oxidized, into the characteristic purple colouring matter haematein, $C_{16}H_{12}O_6$.

This forms violet or purple compounds with copper salts and with alum, and is sometimes used as the basis of inks, which are accordingly termed ***haematein inks***.

Some of the older formulae for iron-gall inks include a small addition of logwood. This was recommended by Lewis (1763), and its advantages have frequently been confirmed since then. Logwood resembles tannin in giving a coloured compound with iron salts, and the greenish-black ink thus formed gradually changes to black on exposure to the air. Several inks made on these lines have been described, but are not altogether satisfactory. Another type of logwood inks consists of the dyestuff with alum and copper, and some also contain a small amount of a chromate.

The formation of an ink by adding a small proportion of potassium chromate to a decoction of logwood was discovered in 1847 by Runge, and since then the reaction has been extensively used for the production of a cheap ink, which is ready for use almost immediately after its preparation. An objection to this type of ink is that it is liable to coagulate in the bottle. Potassium bichromate behaves with logwood in a similar manner to the chromate; and an ink made from logwood extract, potassium bichromate, chrome alum, and oxalic acid is extensively used as a school ink in Germany.

In this country, logwood writing inks are made by one or two manufacturers, and many of the copying inks have a logwood basis.

VANADIUM INKS

Ammonium vanadate behaves like gallotannic acid in forming an ink with iron salts, and has therefore often been recommended as a constituent of inks.

But, although the ink is of a rich black shade, it is not very permanent in the bottle, and the writing done with it gradually turns yellow on keeping. Notwithstanding this drawback, however, the use of vanadium has been claimed in various patents for inks.

ANILINE BLACK INKS

In 1867, solutions of the aniline dye *nigrosine* were first sold under the name of "stylographic ink," for which purpose they are in some respects particularly suitable. As regards permanency, however, they are inferior to properly made iron-gall inks, and can often be removed from the surface of the paper by sponging with water. A solution containing 1·25 per cent of the dye gives an ink of sufficient intensity.

Blue-black inks can also be imitated in aniline colours, and an ink of this type, sold under the name of *induline ink*, was awarded a prize at Paris in 1867.

A similar ink consists of a solution of 1·2 per cent of Bavarian blue, 1·3 per cent of acid green, and 1·5 per cent of chestnut brown. This appears blue-black when applied to the paper and dries to a greyish-black.

COLOURED WRITING INKS

Several allusions are made in the Greek and Roman classics to coloured inks, and we owe the origin of the word " rubric " to the use of the red inks, composed of vermilion, red lead, or red ochre, for illuminating old Roman manuscripts.

Ink of a purple-red shade was also obtained from the shell-fish *Murex*, but, like other red inks, was reserved for the use of the Byzantine emperors.

Green ink is of frequent occurrence in Latin manuscripts, but instances of the use of yellow or blue ink are much less common. At a later period, gold and silver inks were used.

Various methods of preparing coloured inks are described by Canneparius in his book on inks (1660), and these were afterwards supplemented as new soluble dyes were discovered.

Prior to the discovery of alizarine and aniline (1858), coloured inks consisted of preparations of dyes, such as Brazil wood or cochineal for red inks ; a solution of verdigris in vinegar for green inks ; indigo for blue ink ; a decoction of Persian berries for yellow ink ; and logwood mixed with copper acetate and alum for purple ink.

With the exception of logwood ink, these older inks have now been practically replaced by inks made from aniline dyes, the use of which for coloured inks was patented in this country by Clark in 1862.

For instance, eosine, which was discovered by Caro in 1874, is now widely employed in dilute solution as a red ink in place of cochineal. Other red dyes suitable for the purpose are Ponceau scarlet and cotton scarlet.

The blue aniline dyes commonly used in ink making are indigo carmine and soluble blue ; whilst Neptune green and diamond green form green inks ; methyl violet and acid violet, violet inks ; and fast yellow and tartrazine, yellow inks.

Few of the old coloured inks could compare with iron-gall inks for permanency, and this remark applies still more to the aniline inks, some of which, and notably those containing eosine or methyl violet, are particularly fugitive when exposed to the action of air and light.

COPYING INKS

All iron-gall or logwood inks have the properties of yielding an impression to moist paper for a short time after being put upon the paper. In the case of iron-gall inks, for instance, this is due to the fact that the iron tannate which forms when ink dries upon paper is at first soluble in water. Then, as the air gradually acts upon it, it slowly changes into a form which is of a gelatinous nature, and finally into a form which is quite insoluble in water, and only dissolves with difficulty in very dilute acid. The change from the first to the second stage is nearly complete in a day or so, whereas the change into the final stage usually requires many months before it is ended.

In preparing copying inks, advantage is taken of this behaviour of iron-gall and logwood inks. The amount of the ingredients is largely increased, so as to have sufficient pigment in the original writing for transference to one or more copies. At the same time, an increased quantity of an adhesive substance, such as gum arabic or dextrin, is added to keep the pigment on the paper ; whilst a small amount of glycerin or sugar is a common constituent, its object being to retard the first stage of the drying process mentioned above. The presence of too much glycerin, however, is objectionable, as it causes the writing to smudge.

Most of the violet-black copying inks on the market consist of concentrated logwood inks of the types already described, for the logwood extracts usually contain a considerable amount of gummy constituents, which help to retard the drying process.

In the case of aniline inks, the chemical change which occurs with iron-gall and logwood inks does not take place, and copies can, therefore, be taken from the writing at any time afterwards. Coloured copying inks are prepared from the same kinds of dyes as are used for aniline writing inks, but the solutions are made more concentrated, and some sugar or similar substance is added.

COPYING INK PENCILS

Invention of Copying Ink Pencils. It has been pointed out that aniline inks, unlike iron-gall or logwood inks, will yield a copy to moistened paper at any time after they have been applied to paper. It was a further application of this idea to incorporate an aniline dye with the graphite of a pencil and so obtain a mixed pigment, the writing done with which only required moistening to be capable of yielding a good copy.

The first reference in scientific literature to the preparation of this kind of pencil appears in a German chemical journal in 1875, but the invention was patented in this country by Petit in 1874.

Although these pencils were originally intended for copying purposes, it is probable that at the present day they are rarely, if ever, used for that purpose, but are now extensively sold under the name of "indelible pencils" for producing writing which is much less easy to remove than the graphite of ordinary lead pencils.

Composition of the Pigment. The pigment consists of a mixture of graphite, China clay, and an aniline dye incorporated into a uniform mass and compressed to form the core or "lead" of the pencil. Methyl violet is the dye most commonly used, but other aniline dyestuffs and coloured pigments are also employed.

The differences in hardness and colour of the pigments are obtained by varying the proportions of the three main constituents. For example, a hard copying pencil made by Faber of Bavaria contained 25·5 per cent of methyl violet, 24·5 per cent of graphite, and 50·0 per cent of kaolin clay; whilst a soft pencil, made by the same firm, contained 50·0 per cent of methyl violet, 37·5 per cent of graphite, and only 12·5 per cent of kaolin clay.

Speaking generally, the greater the proportion of clay, the harder the pigment and the lighter its colour. There are at least thirty different types of these pencils on the market, and most of them show pronounced differences in the characteristics of the marks made by them on paper.

A series of analyses of the pigments made by the writer has shown that in most cases approximately equal proportions of the three ingredients are used. In another group the kaolin is largely in excess of the graphite, whilst in the case of a few makes of pencil the graphite is largely in excess. Occasionally the clay is altogether omitted, and the pigment then consists only of graphite and dyestuff, and makes a mark on paper resembling that of a blacklead pencil, although the violet dye becomes evident when the writing is moistened.

Copying Properties. As these pencils contain variable proportions of soluble dye, they also vary considerably in their copying capacities ; but, with the exception of one or two kinds, most of them give writing which will yield a very fair copy when pressed with moistened " flimsy " paper in a copying press. The well-known " Swan " pencil, made by Faber, however, contains alumina in its pigment, and makes a very bright mark on paper, but does not give a good copy, although for the same reason its " indelibility " is increased.

Permanence of the Pigment. The question of the permanence of the markings made by copying ink pencils is a matter of some importance, in view of the fact that they are so extensively used as substitutes for writing ink. It has already been mentioned that methyl violet, the favourite dye for these pencils, is particularly fugitive when exposed to light and air, but when mixed with the graphite and clay of these pigments it appears to be decidedly more permanent than when used in printing ink.

The " Membland " Case. Interesting evidence in support of this curious fact was afforded by the case of *King* v. *Macbeth,* which was tried before the King's Bench in 1916. The question at issue was which of two firms of underwriters was responsible for paying the insurance money on the s.s. *Membland,* presumably lost at sea in 1915. If the vessel had been torpedoed, the loss would have been borne by one firm ; whilst if it had fallen victim to the ordinary risks of the sea, the other firm would have been liable. The ship had left Hull in fairly good weather on a coasting trip, but never reached her destination. Some three months later, a wooden stave was picked up on the beach at Hornsea, and on this were written in copying ink pencil the words: " *Membland* torpedoed, port side ; good-bye, dear."

If this stave were genuine, it would prove that the vessel had been sunk, like so many others, by one of the German submarines which were known to have been in those waters.

Although the writing closely resembled that of one of the members of the crew, it was not recognized as his by his mother ; whilst, on the other hand, the relatives of several other members of the crew claimed that it was the writing of their husband or brother, as the case might be.

A subsidiary point was whether it was possible for the writing in copying ink pencil to be exposed to air and light in sea water for a period of three months without being obliterated. Experiments were, therefore, made by the writer to determine whether this was inherently possible, and for this purpose strips of oak, upon which words were written with four different kinds of these pencils, were floated on salt water in a dish which was constantly shaken and frequently exposed to the air and sun. The writing was still distinctly legible in each case after three months, and there could, therefore, be no reason for rejecting the message on the stave on this ground.

In the upshot, the judge stated that, although the stave might possibly be genuine, he was not convinced on the point ; but he decided for other reasons that the vessel had been lost through risks of war.

Identification of Pigments. Owing to the variations in the composition of the pigments, which have been mentioned above, it is possible, under favourable conditions, to distinguish between the writing done with different kinds of pencils. For example, in some cases the violet dye can be rapidly taken up by a solvent, whilst in other cases little, if any, dye can be extracted. Then chemical tests for traces of iron and other substances can be made, and comparative bleaching tests can be followed and the results compared under a microscope.

The Wood Case. By the aid of tests of this kind, it was possible to prove in the case of *Rex* v. *Wood,* tried in 1907, that the writing on certain fragments of paper was in the same kind of pigment as that in a particular pencil. It will be recalled that in that case a woman was found murdered in a house in Camden Town, and that, in the grate, fragments of a burned card were discovered upon which words were written in copying ink pencil. Reproductions of these fragments were published in the Press, and the writing upon them was recognized as that of an artist named Wood. When arrested, this man was found to have upon him a " Swan " copying ink pencil. He denied having written the words on the fragments, and it was, therefore, a matter of importance to determine whether the pigment in his pencil agreed in its characteristics with the pigment in the writing.

Comparative tests made at the same time showed that there was an exact agreement between the two pigments in colour, in copying properties, and in chemical reactions, whereas similar tests applied to the pigments of a number of other typical copying ink pencils gave very different results. Evidence to this effect was given at the trial, and ultimately Wood admitted that he had written the post card in question. He still denied all knowledge of the murder, and in the end was found not guilty. A full account of this case will be found in the writer's book *Science and the Criminal.*

Age of Writing in Copying Ink Pencil. In the case of writing ink, it is sometimes possible to form an opinion as to the approximate age of the ink in writing, owing to the gradual nature of the chemical changes which occur. (*See* p. 56.) In the case of copying ink pencil pigments, however, this is not possible, since, with the exception of some fading of the dye, which will vary with the conditions, the pigment remains in the same state from the time when it is first applied to the paper.

The deductions as to prior writing, which can sometimes be drawn when lines written in ink (*see* p. 55) or blacklead pencil cross each other, are only exceptionally possible in the case of writing done with copying ink pencils, for the dyestuff conceals the lines made by the graphite or clay portions of the pigment.

The question of the age of the entries in a rent book which had been written with a copying-ink pencil was raised in the Seddon trial ; but, although all the entries could be proved to be written with a pencil or pencils containing the same kind of pigment, there were no data available upon which any opinion as to the approximate date of any portion of the entries could be based.

VI

CHARACTERISTICS OF INK IN WRITING

THE differences in the nature and composition of various kinds of writing ink have been outlined in the preceding pages, and it also may be of interest and of practical value if a brief account is given of the characteristics of the various inks after they have been applied to the paper.

Colour of Ink in Writing. When examined under a microscope, the layer of pigment in ordinary blue-black writing ink will appear as a bluish or dark violet deposit, which is broken up by the fibres of the paper so as to give a sort of crystalline effect. The colour will depend largely upon the length of time that has passed since the ink began to dry, as well as upon the nature of the particular dye that was in the original ink. It is also greatly affected by the action of blotting, and by the duration of the period between writing and the application of the blotting paper. The reason for this is that the formation of the true ink pigment, the insoluble tannate of iron, begins immediately after the ink is put on to the paper ; and that the sooner the blotting paper is applied, the larger the proportion of potential ink pigment removed. Hence, a delay of even 10 seconds in applying the blotting paper may make a very material difference in the colour of the final writing. The sooner the writing is blotted, the paler and the bluer will it be when dry, for the blue dye in the ink acts upon the paper much more quickly than the iron and tannin ingredients, which are converted by the oxygen of the air into the insoluble tannate forming the black pigment of the ink.

It is, therefore, as a rule, not possible to draw any definite conclusions as to the age of the writing from a single examination of the colour of the ink. This was the mistake that was made in the case of *Rex* v. *Cohen*, in which a doctor was accused of drugging a man to keep him out of the Army. His reply to the charge was that the man was a regular patient, and he produced his ledgers, in which there were numerous entries of the name of this man. Owing mainly to the fact that these entries were all in much paler ink than adjoining entries, it was suggested by the Crown that they were false and had been inserted at a later date, and Dr. Cohen was sentenced to a fine of £100 and six months' imprisonment. He appealed against the sentence, and at the hearing of the appeal it was clearly shown that the paleness of the ink could not be regarded as a proof that the disputed entries had been more recently inserted than those preceding or following them. There was also positive evidence to the contrary in the fact that in the case of one of the entries, portions of the lines came in contact with the top of the writing of the following entry, and could be shown to be below, not above, the lines of that second entry ; whereas if the disputed entry had been inserted afterwards, these portions of the line would have been on top. These points, taken in conjunction with other evidence, were sufficient to quash the sentence upon the defendant.

The Drying of Ink. In the so-called " drying " of iron-gall ink upon paper, a somewhat complicated change takes place, the nature of which is mentioned in the section dealing with " Copying Inks." In the original ink, the true pigment must be regarded as in an unformed condition, although, as has been pointed out, in the older type of inks the change completed by " drying " on paper was allowed to proceed a long way on its course in the ink itself to prevent the liquid being too pale. If an iron-gall ink, free from acid or added dye, is exposed to the air in an open vessel, it will slowly darken to a violet-blue liquid, while simultaneously a black crust will be formed on the surface. This layer of solid ink, when first formed, is somewhat soluble in water, but becomes more insoluble on standing, and eventually, after a long period, does not dissolve readily in dilute acids.

In all probability this series of changes is what takes place when the ink dries on paper, and will explain the behaviour of the ink in the copying process and on treatment with dilute reagents as a method of estimating its age. For a very short time after its application to the paper, ink can be washed off the surface without leaving any trace behind. The pigment, which is somewhat soluble in water, is then formed; and so long as the change has not proceeded much further than this, it will dissolve when pressed with moistened absorbent paper. This period, which may be measured in hours or days in the case of ink not specially prepared for the copying process (*see* p. 47), will vary according to the character of the ink, the amount applied to the paper, and the conditions to which the writing has been exposed.

Estimation of the Age of Ink in Writing. Attempts have been made to base methods of estimating the age of ink in writing upon the possibility of obtaining copies from it under specified conditions; but, except under very exceptional circumstances, it is not possible to place much reliance upon the results.

The second stage of the change from the partly soluble compound into the compound which resists dilute acids, is a very gradual one, and may not be quite complete until after the lapse of three or four years, or even longer. The iron tannate will then be of a more resinous character, so that it repels the attack of dilute acids or of bleaching agents. Here we have a change which may be of much more value as a means of judging of the age of the ink, provided that allowance is made for the long period required to complete the change. As an illustration of the use of this method, the case of *Rex.* v. *Pilcher*, an account of which is given in the writer's book *Science and the Criminal*, may be cited. In this case, Colonel Pilcher had produced a will, dated eleven years previously, which had made him the heir to his cousin's property, and on the strength of this will had taken possession of the estate. The will was disputed by relatives, who alleged that it was a forgery fabricated at a much later date than that stated upon it.

Now, in the case of writing in ordinary iron-gall ink on a document eleven years old, the change into the relatively insoluble iron tannate would normally have been completed several years, and the writing would only react very slowly with dilute acids and other reagents. Permission was, therefore, obtained from the President of the Probate Court to apply a series of tests of this kind to the inks upon the alleged will. As soon as the reagents were applied to the writing of the body of the document, and to the alleged signatures of the testatrix and witnesses, the reaction in each place took place immediately, the ink spreading in blue smudges over the paper. This proved beyond a doubt that a change into the final iron tannate had not occurred in any of the four inks upon the will. On the other hand, similar tests applied to the inks upon a series of cheques, dating back for more than ten years, showed that intense reactions were only obtained for about three years, and that after five or six years the cheques gave but little indication of a reaction, and none at all after eight years.

Analogous results were obtained with other specimens of old writing of the deceased lady, and the pronounced differences between these and the results given by the will justified the conclusion that that document could not be as old as it purported to be, and that it had probably been written within the preceding four or five years.

Even after evidence to this effect had been given, Colonel Pilcher continued to assert that the will was genuine ; but ultimately, before the end of the trial, he made what the judge termed a half-confession, admitting that he had uttered the will knowing it to be a forgery, but that he did not know who was responsible for the forgery.

Carbon Inks in Writing. It is a simple matter to distinguish between the ancient carbon inks that were used on Egyptian papyri, and which are still in use in the East, and the iron-gall inks which succeeded them. As carbon is not affected by any of the dilute reagents which will bleach ordinary writing ink, all that is necessary is to apply a reagent of this type and to examine the place under the microscope. If a carbon ink was present, the undissolved black particles of carbon, due to the lampblack or other pigment, will still be discernible after the treatment.

It is also possible, by means of analogous tests, to distinguish between ordinary writing and Indian ink, printing ink, lithographic ink, and those kinds of typing ink containing lampblack or gas black. In this way, the writer was able to prove that a plan, long believed to have been drawn by Nelson, was really only a lithographed copy.

Sometimes the ink is a mixture, as in the case of " safety inks " (*see* p. 118) to which carbon has been added, and the writing done with such a mixed ink will be partly bleached ; whilst the carbon particles will be left unattacked, as before, although usually much more scattered than in the case of an all-carbon ink. Sometimes a carbon ink has been added to an ordinary iron-gall ink with the object of making the writing look old. Thus, a clerk who was accused of fraud confessed that he had falsified an entire ledger in this way by adding Indian ink to blue-black writing ink to make recent entries appear old. The writing in question had a brownish tint and appeared older than writing of the same date written in blue-black ink. Chemical and microscopical examination of these entries confirmed the statement, for, after the iron-gall ink had been bleached, fine black particles of carbon could be seen under the microscope.

Modern Inks in Writing. The various classes of writing inks described in the preceding pages can be distinguished from one another by means of chemical tests. For example, all the iron-gall inks will give pronounced reactions for iron which will not be given by logwood inks or by aniline black inks. These two last classes may also be readily differentiated by other reactions, such as treatment with dilute acid or bleaching solutions. The presence of the added blue dye, which still remains in dried ink in the writing, readily enables blue-black inks to be distinguished from the older type of iron-gall inks. On treating the writing with a dilute acid, the iron tannate is bleached, while the dye is left, so that the writing becomes bright blue. Aniline blue dyes have been added to writing inks for upwards of fifty years, but the presence of such a dye in the ink in entries in old family Bibles has, on more than one occasion, proved fatal to a claim for an old-age pension.

Differences in Blue-black Inks. The problem of distinguishing between different makes of blue-black ink in writing is a much more difficult one than that of separating the inks into different classes. Sometimes it is necessary to determine whether the whole of the writing on a document is written in the same kind of ink, or whether a particular piece of writing is in the same kind of ink as another piece of writing. The tests upon the results of which answers can, under favourable conditions, be given to questions such as these are based upon the facts that ink makers use different blue dyes and different proportions of those dyes in their inks, as well as different quantities of iron salts and of tannin from different sources. All of these factors will have an influence upon the behaviour of the reactions, and by making a series of tests under the same conditions it is frequently possible to distinguish between blue-black inks in writing which, to a casual observer, would appear practically the same. The principle may be illustrated by reference to the first occasion on which evidence of this kind was given in an English Court of Law. In the Brinkley poisoning case (1907), the motive suggested for the crime was that of attempting to get rid of a man named Parker who had refused to corroborate the statement that he had witnessed a will. He admitted having signed a paper at a public-house, but asserted that he had been told that his signature was wanted to a petition for an excursion. Brinkley, who had taken possession of the property in virtue of this will, determined to get rid of his uncompromising " witness," and left a bottle of poisoned stout in Parker's lodgings. This was drunk, not by the intended victim, but by his landlord and landlady, both of whom died ;

and inquiries led to the arrest of Brinkley, who was charged with the murder. In order to test the accuracy of Parker's statement, the bottle of ink was obtained from the public-house and the ink in it compared with that of Parker's signature upon the alleged will. The dye contained in this ink was of a particularly brilliant hue, and writing done with it and left for a few days exactly matched that of the signature. There were also two other inks upon the will, the body of which and the signature of the alleged testatrix were written with one kind of ink, whilst the signature of the second witness was in another ink. Brinkley did not contradict this evidence, but attempted to explain it by asserting that the old lady whose will he had forged had three different kinds of ink in the house. Only one of these was forthcoming, however, and that agreed in its characteristics with the ink in the writing of the body of the will. The prisoner was found guilty of the murder.[1]

The Fading of Ink. After iron-gall ink has attained its maximum of colour in the drying process, and the formation of the insoluble tannate is complete, it normally remains constant in its characteristic for many years, with the exception of more or less browning due to the action of the air upon the iron compound. This stability, however, depends upon the presence of sufficient amounts of iron and of tannin, and upon the quantities being in the correct ratio with regard to each other. If either the iron or tannin is materially in excess, the ink in the writing will be much less permanent, and this is one of the reasons why old writing is sometimes of a pale yellow colour, for the tannin constituent may then have completely perished, leaving only the iron in the form of an oxide. In such cases, it is possible to restore faded and nearly invisible writing by treating the place with an acidified solution of potassium ferrocyanide, which will react with the residual iron and reproduce the original characters in the form of Prussian blue or a similar compound. The blue aniline dyes added to blue-black inks as provisional colours are, in the case of a well-made ink, much more fugitive than the pigment or iron tannate, although they offer more resistance to the action of dilute acid and other chemical agents. If, however, the writing has been kept protected from light, a blue dye can still be detected after many years. For example, the writer has found such a dye in writing that was more than forty years old. The presence or absence of residual traces of bleaching agents or other chemicals in the paper undoubtedly has a considerable influence upon the permanence of the writing in iron-gall ink, and the use of a standard paper free from chlorine or acid substances is as essential for documentary purposes, as is an ink of the proper composition.

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[1] For further particulars of this case, see *Science and the Criminal.*

Although carbon is admittedly one of the most permanent pigments, it is remarkable that the writing in carbon inks in some of the old MSS. has faded from what was, presumably, a rich black to a dull brown.

Mr. A. Lucas, Director of the Government Laboratory in Cairo, has investigated several instances of the kind, and has come to the conclusion that the fading must be attributed to the use of an impure carbon, such as that obtained by charring date stones, in the preparation of the ink. The carbon inks on ancient MSS. frequently give a distinct reaction for iron, which may have played a part in the browning process. This iron is, in all probability, due to impurities in the carbonaceous material used as the carbon of the ink ; and there is no justification for concluding, as Underwood did in 1857, that the presence of iron in carbon inks on paper is evidence that a mixture of carbon and iron-gall inks has been used.

Erasure of Inks. In the days when vellum was the principal medium for writing, it was not an uncommon practice for the scribes in the monasteries to erase former writing from old MSS. by rubbing the surface with pumice stone and to use the cleansed surface for further writing. Such rewritten MSS. are known as palimpsests (from *palin* = again, and *psestos* = rubbed), and there is little doubt that many of the lost writings of classical times were in this way defaced with worthless commentaries of the monks. The most striking instance of the kind is, perhaps, the discovery of the *Institutes* of Gaius, the classical Roman lawyer. It had long been known from references in contemporary writings that such a treatise had been written, but all traces of any copy of the work had been lost until 1816, when Niebuhr, the German historian, discovered the greater portion of the work in the under-writing in a palimpsest in the chapter-house at Verona. The methods of deciphering such twice-written documents depend upon the facts that iron-gall inks penetrated fairly deeply into the membrane of the vellum, and that the abrasive process, used to remove the prior writing, did not effect a complete removal, but scraped the surface, still leaving some of the iron of the ink within the fibres of the skin. Hence, by examining the document with the light reflected upon it obliquely, it is sometimes possible to read the original characters.

Modern applications of photography have proved of great value in deciphering the earlier writing on palimpsests and, by a combination of negative and positive prints, obtained with the aid of light filters and colour sensitive plates, results have been obtained in some cases which would not have been possible by any other optical method.

Re-development of the erased writing, by the application of chemical reagents, is, when permissible, an effective method ; but it has the drawback of staining the surface of the MSS., and should only be applied when photographic methods have failed. The principle upon which such chemical treatment depends is essentially the same as that described above in reference to faded writing, the practically invisible iron in the prior writing being treated with a reagent that will combine with it and form a dark compound of iron (Prussian blue or the like) which can be readily seen.

In order to avoid injury to valuable documents by the use of liquid chemicals, various methods have been devised in which the MS. is moistened with water and fixed face downwards in a closed box over a receptacle containing a volatile reagent, the vapours from which will gradually act upon the traces of iron in the original ink and form a compound that will make the characters legible.

Mechanical Erasure on Paper. Inks which have been effectively removed from paper by scratching the surface with a knife, seldom leave more than faint traces of iron in the paper ; and, as a rule, there is little prospect of restoring the original writing by development with chemical reagents, or of deciphering the characters by photographic devices in the manner which has given such good results with erased writing on vellum.

The question usually arises in connection with the fraudulent erasure of words or figures in modern documents, and in such cases it is often a matter of importance to be able to demonstrate the fact that an erasure has been made, even though it is not possible to discover what was originally present.

As mechanical erasure removes the sizing from the surface of the paper and also renders the paper itself thinner at that place, it may be possible to demonstrate by means of a photograph that the document is more transparent at that point than elsewhere. Moreover, the removal of size causes the paper to be more absorbent where it has been scratched than on other parts of the surface, and this may be indicated by applying a coloured solution to the paper.

Certain chemical reagents, such as iodine vapour, are also useful upon occasion for proving that some alteration or other has taken place on the surface of the paper.

Chemical Removal of Ink. Any writing ink not containing lampblack or other form of carbon can usually be bleached by means of a suitable reagent, such as an acidified solution of bleaching powder, so that little, if any, trace of the original writing can be seen. The erasure is not always so complete, however, as at first sight it appears to be, and the obliterated characters can sometimes be detected by means of photography, with the aid of colour-sensitive plates and screens.

In the case of iron-gall inks, much of the iron constituent of the ink will, as a rule, still remain in the paper, although the iron compound will be very pale yellow and will thus escape notice, especially on a cream-tinted paper. By treating the suspected place with a reagent for iron, in the manner described above, it may frequently be possible to restore the original writing.

A case of this kind recently came within the writer's experience. A confidential clerk paid in a large sum of money to the credit of a particular firm, and this amount was entered on the counterfoil of the paying-in book. He then bleached the ink in the written name of this firm by means of a chlorine solution, and substituted for it the name of his own firm. The bank, believing that a mistake had been made, made the necessary alteration in their books, and the clerk was then able to withdraw this sum by a cheque which he signed on behalf of his own firm. The prior writing on the counterfoil could not be seen on casual inspection, although by closely studying the place with various lightings, faint indications of pale straw-coloured markings could be discerned, and these were somewhat plainer in an enlarged photographic print.

Chemical tests, however, gave more satisfactory results ; and by carefully brushing the spot first with a very dilute acid, and then with a reagent for iron, the original characters were restored, and the prior words then appeared as dark blue writing intersecting the later writing on the counterfoil.

The Ink on Charred Documents. Even when a document has been burned so as to leave only a thin film of charred paper, the ink in any writing upon it need not necessarily be destroyed.

The possibility of deciphering the characters, however, will depend both upon the nature of the ink and upon the degree of coherence of the charred mass. If the paper has been completely calcined to a black mass, any writing in aniline ink will have disappeared ; but an ordinary iron-gall ink will leave a residue of the iron in the form of an iron oxide or sulphate, and this may sometimes be deciphered by photographic methods.

Printing ink is also discernible after burning, partly on account of the fact that the lampblack it contains does not burn so readily as the charcoal formed by the paper, and partly owing to the presence of mineral constituents, such as Prussian blue, in its composition.

Writing in copying-ink pencil or in ordinary pencil usually remains visible for the same reason, the graphite in the pigments of such pencils being very difficult to burn away completely.

A method of rendering the writing upon charred paper more visible is to immerse the fragments in solutions of salts, such as those used in the preparation of gas mantles, and then to calcine them in such a way as to leave a coherent white ash, upon which the mineral residue left by the ink will then be clearly seen.

Fragments thus treated are preserved for further examination between two sheets of glass, or they may be treated with collodion solution, like gas mantles, and will then be flexible and suitable for careful handling without the protection of glass plates.

MARKING INKS

Natural Marking Inks. There are many natural vegetable juices which can be used for making permanent marks on textile fabrics, and some of these have long been used for this purpose.

Among the plants which produce juices darkening on exposure to the air may be mentioned *Rhus toxicodendron*, the poison ivy of North America, a large shrub, the leaves of which are used commercially in the preparation of a black stain. The milky juice from the leaves slowly blackens on contact with the air, and was recommended for use as a marking ink early in the eighteenth century. The juice has an irritating action on the skin, whence its name " poison tree."

Rhus venenata, the " poison sumach," is the source of a similar varnish ; whilst an analogous species, *Rhus vernicia*, is used for the same purpose in Japan.

The Marking Nut. The nuts produced by the Indian tree, *Semecarpus anacardium*, have a mesocarp made up of large cells, within which is secreted a sticky brown juice, which, when mixed with lime, will produce a permanent mark upon textile fabrics. The commercial product has the appearance of the examples shown in Fig. 13, which represents two forms of the nut and a vertical section showing the construction of the ink secreting cells.

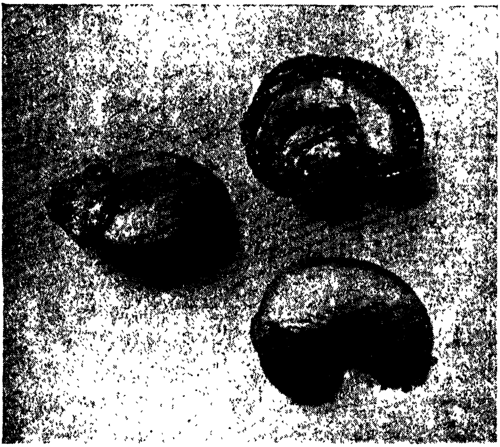


FIG. 13

INDIAN MARKING NUT

The section shows the pigment cells

The nuts, which are also obtained in the West Indies and in Australia, are used in the preparation of a black stain and probably as secret ingredients of marking inks. They were recommended for this purpose by Lewis as long ago as 1763. Marks made with the prepared juice on linen resist the action of several powerful chemical agents, but can be removed by treatment with ether.

Silver Marking Inks. The principle upon which the well-known silver marking inks are based is that when a silver salt is exposed to the light, it is reduced and a black oxide of silver is left. Other instances of this phenomenon are to be seen in the darkening of caustic silver on the skin, and in photography.

Originally the fabric which was to be marked was first prepared by treatment with a mordant known as a *pounce,* the object of which was to convert the silver salt, usually silver nitrate, into a form in which it would be readily reduced by the light.

This type of marking ink was afterwards superseded by inks containing silver tartrate, which were reduced to the black oxide when a hot iron was passed over the writing. Many of these preparations were based upon the formula published by Redwood. These inks contain a certain proportion of gum arabic or gum tragacanth to prevent their running on the fabric, and are coloured with sap green or archil, as a temporary colouring matter, to make the writing visible before the ironing.

An ink on these lines was afterwards patented by Reade (1846). In some cases, platinum or gold salts were also used in marking inks, but at the present day would obviously be too expensive.

In addition to silver marking inks, preparations containing bichromates, copper or manganese salts, and molybdic acid have also been described and in some cases patented, but none of these ever obtained the popularity of the silver tartrate inks, which are still extensively used.

Aniline Marking Inks. The chief competitors with the silver inks have been the aniline black inks, since they do not require any application of heat to the writing.

One of the earliest of these inks was that invented by Jacobsen in 1867. Like all the early aniline inks, this was prepared in two solutions, which, when mixed, reacted together and formed aniline black within the fibres of the fabric. One of the solutions contained aniline " salts " (i.e. aniline hydrochloride), with gum arabic and glycerin. The other contained an oxidizing agent composed of copper chloride, sodium chlorate, and ammonium chloride.

Writing done on linen with a mixture of the two solutions was green at first and then changed to black, the change being promoted by heat, although sometimes this rendered the fabric brittle and made holes.

Many modifications of this formula have been made and patented, and various ingredients as, for instance, paraphenylene-diamine, have been introduced to intensify the blackness of the writing. Essentially, however, the two-solution aniline inks on the market all depend upon the same principle of the formation of aniline black within the fibres.

One-solution Aniline Inks. The drawback of having to mix two solutions before using the ink led to a number of experiments to discover a means of keeping the ingredients in one solution under such conditions that they will not react until the liquid is applied to the fibres.

The problem has been successfully solved by several makers, but their processes are carefully guarded as trade secrets. In some of these preparations an excess of aniline oil is present ; in others, acetic acid ; and the liquid remains stable in the bottle so long as it is kept protected from the air. When applied to the fabric, however, the excess of the particular substance evaporates, and the other ingredients then react together to form aniline black, just as in the case of the two-solution inks.

Indigo Marking Inks. Blue marking inks, based on a similar principle to that used in the preparation of aniline marking inks, have also been put on the market. The blue colouring matter of indigo is termed *indigotin* in chemical language. When treated with a certain class of chemical agents, known as reducing substances, it is decolorized, and is then known as *indigo white.* This dissolves readily in alkali solutions, and this colourless liquid, when exposed to the air, is gradually oxidized and the original blue compound is again formed.

In one of the published formulae for an ink of this type, a mixture of indigo blue and copperas (as reducing agent) is treated with a solution of caustic soda in a closed bottle. After a few days the liquid becomes colourless, through the formation of the indigo white, and is then decanted and mixed with a small amount of gum mucilage and a little litmus (as a provisional colouring matter). When this alkaline liquid is used on a fabric as a marking ink, it is gradually acted upon by the oxygen of the air, and indigo blue is re-formed within the fabric. The objections to this ink are that, owing to its pronounced alkalinity, it is liable to injure the fabric, and that the re-oxidation of the indigo white to indigo blue will also take place gradually in the bottle, and give rise to a blue deposit.

Another blue marking ink, patented in 1880 by Johnson, also depends upon the formation of indigo blue in the fibres of the material. A solution of *o*-nitrophenyl-propiolic acid, with a reducing agent such as glucose, and an alkali substance, is used as the ink; and the writing is then developed by steaming the marked portion of the fabric. This leaves the marking in blue characters, whilst if the fabric is exposed to dry heat the writing becomes black.

Red Marking Inks. The principle of fixing a dyestuff upon the fibres of a fabric by treatment with a suitable mordant was patented in 1864 by Möller. The material was first prepared by treatment with alum, and the marking then done with an ink composed of an extract of madder mixed with an alkali salt and suitably thickened with gum, and containing some vermilion as a provisional colour.

In other patent marking inks, dyes are used in admixture with a solution of rubber (Langbeck, 1882), or dyes soluble in oils are dissolved in castor oil and the solution thinned with oil of turpentine (Hickisson, 1893).

A French red marking ink is made by dissolving Congo red in a hot dilute solution of caustic soda and glycerin and thickening the mixture with gum. Ink of this type is suitable only for temporary marking, and will not resist frequent washing.

Methods of Examination.· The principal marking inks met with in commerce are the black silver inks and the two-solution and one-solution aniline black inks, and these are readily distinguished by chemical examination.

The behaviour of the ink in practice is, however, of much more importance than its chemical composition, and in testing such inks it is usual to follow the directions given on the label, and to apply a series of tests to strips of cotton or linen fabric which have been marked in that way.

The first essential of a good preparation is that it shall not make holes in the fabric, even when the directions of the manufacturer have not been closely followed. For example, certain aniline marking inks are liable to " tender " the fibres of the material if the marks are ironed before the fabric is washed; whereas, if the material is carefully steamed after marking, no perceptible injury is apparent.

The next consideration is that the markings shall be of a good rich black, and shall not be removed in the ordinary course of washing. Some aniline black inks give more greenish writing than others.

As tests of permanency, the marked strips of fabric may be boiled for 30 minutes, first with a solution of soap and washing soda, then with a weak solution of bleaching powder, and lastly with a solution of sodium perborate. A good marking ink will not show any indication of fading after this treatment.

It is also necessary that the ink shall be sufficiently fluid to flow from the pen, and yet thick enough not to " run " on the fabric. This factor depends upon the proportion of gum added to the ink.

Lastly, the ink must be reasonably stable. That is to say, it must not become solid in the bottle, or decompose if the bottle is left uncorked for a considerable time.

Simplicity in the manner of using is a minor consideration, although it becomes of more importance when a large amount of marking is required, as in a laundry sorting room. This factor accounts for the preference now given to one-solution aniline inks over the earlier two-solution inks.

Marking Ink Pencils. The idea of combining a marking ink with a pencil pigment suggested itself many years ago, and many pencils, both black and coloured, have been sold at different times as convenient means of marking linen.

In the earliest pencils of the kind, a silver salt was incorporated with blacklead, and the marking was ironed in the same way as a silver marking ink. Patents for the manufacture of such pencils were taken out by Dunn in 1858 and by Schroll in 1877, whilst in 1878 Hickisson claimed the use of a pencil pigment incorporated with a vanadium marking ink.

Coloured marking ink pencils have been made by mixing pigments with various bases to form the " lead " of the pencil, such as, for instance, a solution of an aniline dye in castor oil, with a basis of clay, dextrin, or the like. The objection to coloured inks of this type is also applicable to pencils made from similar pigments, and the writer has not yet met with a coloured marking-ink pencil which will produce writing with any claim to permanence.

VII

INDIAN INK AND OTHER INKS FOR ARTISTS

Indian Ink. It has already been mentioned that inks with a basis of carbon were the earliest kinds of inks used for writing, and that they are still used for this purpose in Egypt and other countries of the East.

In Europe, however, they have been displaced for this purpose by inks made from galls, logwood or aniline dyes, and the so-called Indian or Chinese ink is now mainly used as a drawing ink or as a pigment for artists.

The process of making this product is in all essential details the same as that described by Chen-ki-Souen more than three thousand years ago.

Lampblack is made by burning some form of carbonaceous material with a limited supply of air, and causing the smoke to form a deposit of soot upon a cover placed over it. At one time, rosin, rice straw, beans, or wood were largely used for the purpose ; but for the finest qualities of ink, only a fatty vegetable oil, and especially that now known as " Chinese wood oil," was burned.

The earliest types of Chinese ink, like the carbon ink of modern Egypt, consisted of a liquid preparation of lampblack suspended in a solution of gum or glue, and the solid product was not made until a later date.

The method of collecting the lampblack is represented in the spirited drawing by Chen-ki-Souen (Fig. 14).

The oil is burned with a smoky flame in a series of terra-cotta lamps provided with covers upon which the soot deposits, these covers being cooled by water put into a hollow at the top. Much skill is required to

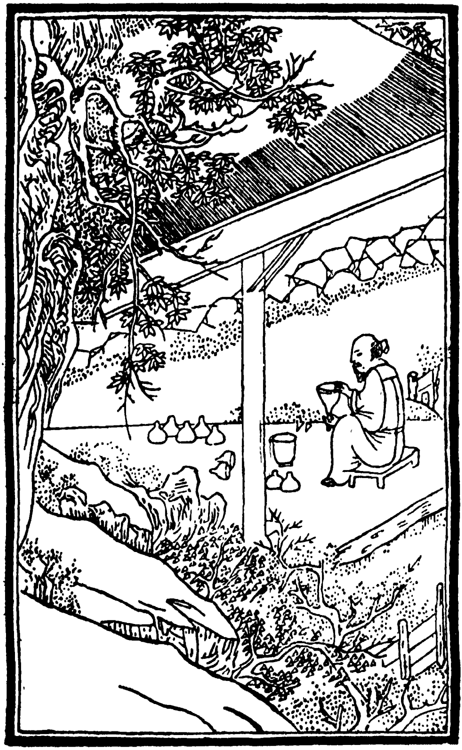


FIG. 14

THE MANUFACTURE OF CHINESE INK

Workman collecting lampblack

judge the right moment to stop the process; if the flame is not properly regulated in relation to the supply of air, or if the lamps burn too long, the black will have a yellow tint (due to oily impurities), and will be suitable only for inferior products.

In a later development of the process, the terra-cotta lamps are grouped in a chamber in which is a ventilating shaft to regulate the supply of air. A method of fractionating the lampblack into coarser and finer grades has also been developed, the smoke from pine wood being conveyed through a series of chambers, constructed of bamboo covered with paper, the deposit in the last compartment being the finest.

The lampblack collecting in the cones is stroked with a feather from the surface into glazed jars, and then sifted and dried in paper bags suspended in a room. It is now ready for mixing with the solution of glue, which is prepared either from fish or from ox-hide, and passed through a sieve on to the weighed quantity of lampblack. (*See* Fig. 15.) The mixture is heated for a short time in a closed vessel over boiling water, and is then pounded for three or four hours as represented in Fig. 16.

Musk and camphor are now added, and the plastic mass is rolled into long sticks, which are moulded into cakes and dried for three or four days in ash from rice straw. A similar process of making Indian ink has long been in use in Japan.

The composition of the commercial products from China does not show very wide variations. Typical samples examined by the writer have contained from 7·2 to 9·9 of water, 49·6 to 57·0 of carbon, 3·69 to 4·96 of ash, and 4·87 to 7·74 per cent of nitrogen. They differ greatly, however, in their behaviour, the best quality, in octagonal sticks, readily yielding a fluid

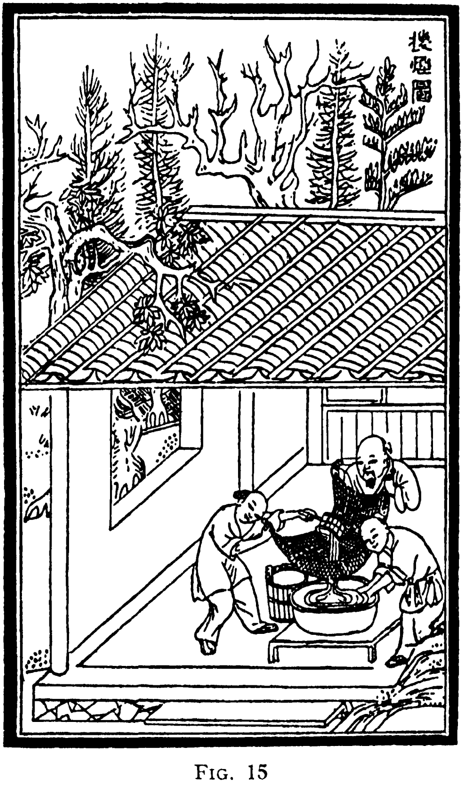


FIG. 15

THE MANUFACTURE OF CHINESE INK

Straining the glue

pigment when rubbed with a little water, and retaining the carbon in suspension for a long time. The better grades are also of a much richer shade of black.

The liquid preparations of Indian ink, sold to artists, are sometimes made by grinding up with water the fragments of solid Chinese or Japanese cakes which have broken in transit.

Another modern product is made by mixing purified European lampblack with a solution of gelatin which has been boiled under pressure until it no longer gelatinizes on cooling, and so is suitable for the preparation of a liquid Indian ink which will not become solid in cold weather.

SEPIA

The well-known pigment sepia is a natural carbonaceous ink secreted by the common cuttle-fish or squid (*Sepia officinalis*) and other species of *Cephalopoda* as a means of enabling it to escape from its enemies.

The pigment or ink which it uses for this purpose is formed in a special glandular organ termed the ink sac, and is so extremely diffusible that it will immediately render opaque a thousand times its volume of water.

It was mentioned in the historical outline that sepia was known to the ancient Romans, who probably used it as a writing ink, but it has long been superseded for this purpose by preparations more suitable from every point of view.

The ink sacs are mainly obtained from species of cuttle-fish caught in the Mediterranean, but considerable quantities of the crude pigment are also imported from Ceylon. Fig. 17 represents a typical specimen, which was kindly sent to the writer by Mr. James Newman.

In preparing the pigment for commerce, the dried

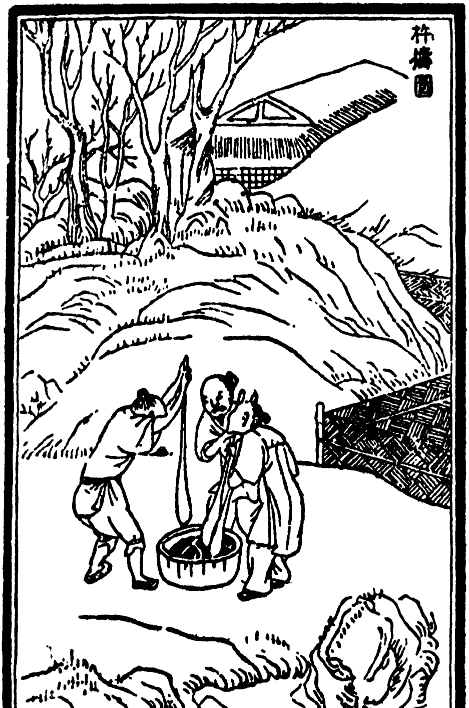


Fig. 16

THE MANUFACTURE OF CHINESE INK

Pounding the lampblack and glue

sacs, which are very hard and brittle, are ground to a fine powder, which is then extracted with a boiling solution of caustic soda. The extract is filtered and the dark brown filtrate is neutralized with hydrochloric acid, which throws down a precipitate of the pigmentary substance, to which the name of *melanin* has been given.

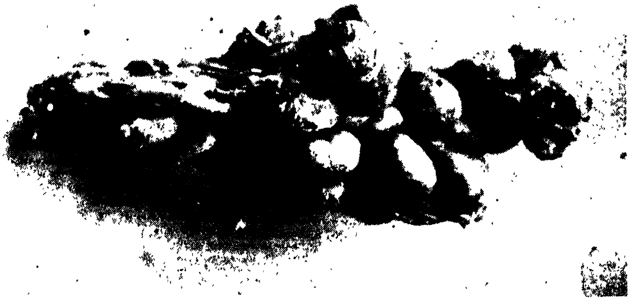


FIG. 17

DRIED INK SAC OF CUTTLE-FISH

About two-thirds natural size. Used as the raw material for sepia

This precipitate is repeatedly washed with water and is dried at a low temperature. Finally, it is ground up into the finest possible state of division, and either compressed into cakes or put up as a liquid preparation, or mixed with oil as an oil paint.

Although sepia is a carbonaceous pigment, it contains, in addition to carbon, other elements, including hydrogen, oxygen, nitrogen, and sulphur. It may, therefore, be distinguished from Indian ink, with which it is sometimes adulterated, by the fact that nitrogen is present in the black pigment ; whereas, in the case of Indian ink, any material amount of nitrogen is present only in the added glue, which can be separated from the lampblack by washing the mixture with water.

It was at one time commonly accepted that sepia was one of the most permanent pigments, but the experiments of Dr. Russell and Sir William Abney have shown that it is a decidedly fugitive colour.

VIII

PRINTING INKS

Historical Outline. There is some evidence to show that a primitive form of printing was in use in China before the Christian era, and at a later period Chinese books were printed from blocks cut in stone. The carbon ink, the preparation of which has already been described, would only require modifying by the addition of an oil varnish to give an ink suitable for this purpose.

In all essential respects, printing ink is a form of paint which dries very rapidly, and it has its forerunner in the *atramentum tectorium*, or ink used in ancient Rome for decorating walls. The printing inks used in the early European block books of the fifteenth century, fine examples of which may be seen in the British Museum, vary very greatly in their present tone, some being extremely black and others a poor brown. This was probably due to the use of an impure form of lampblack for the ink.

The work on inks by Canneparius (1660), to which allusion has already been made, describes a method of preparing printing ink, in which 1 lb. of linseed oil varnish, containing juniper, was mixed with 1 oz. of smoke-black, and the mixture boiled over a slow fire until sufficiently thickened.

In 1683 a book was published by Moxon with the title of *Mechanick Exercises*, in which a description is given of the methods of preparing Dutch and English printing inks, much to the disadvantage of the latter, which are stated to have been insufficiently boiled, so that the oil separated from the ink on the paper.

In the old method of preparing the Dutch ink, old linseed oil was boiled in a closed vessel, and the vapours fired at intervals (after momentarily raising the cover) until the varnish was thick enough for use.

In 1723 a French printer, Fertel, described a similar method of preparing the varnish by burning the oil. The varnish was then thoroughly mixed with smoke-black, made from pitch resin, in the proportion of 2 lb. of varnish to 5 oz. of black. In the recipe of Breton (1751), which became a standard one, nut oil was heated for two hours in a closed iron or copper vessel, and then " burnt " several times, and the heating afterwards continued over a slow fire until the varnish was as thick as glue. It was then strained through linen and mixed by the pressman, on the inking table, with 2½ oz. of lampblack for each 1 lb. of varnish.

The addition of soap as an ingredient of printing inks, to increase the sharpness of the impression and prevent clogging of the type, was first published in 1823, although the practice had probably been in use long before that date. In one of these early recipes, an addition of 2 lb. of black rosin and 1 lb. of hard soap was made to the hot oil varnish.

The formulae upon which the printers of the early part of last century largely relied were those published by Savage, in 1832, in his book *The Preparation of Printing Ink.* There he describes the method of making the varnish by heating six quarts of old linseed oil in a covered iron pot and firing the vapours from time to time until, on cooling, it could be drawn out into strings an inch long. After being " burnt " in this way, the oil was mixed with 6 lb. of rosin and 1¾ lb. of brown soap cut into slices and, while still warm, was poured into a vessel containing a little Prussian blue or indigo, 4 lb. of mineral lampblack, and 3½ lb. of vegetable black. Finally, the ink was thoroughly mixed and worked up in a small mill into a uniform smooth paste.

Savage was a great advocate of the printer's preparing his own inks, especially in the case of coloured inks, much smaller quantities of which would usually be required than of black ink.

Preparation of the Varnish. The old method of " burning " the linseed oil is not yet obsolete, although it has been shown that the thickening effect can be obtained by blowing air into the heated oil.

The principle upon which both the old and newer processes is based is that a certain class of oils, typified by linseed, hempseed, and walnut (nut) oils, contain a relatively large proportion of highly unsaturated fatty acids in combination with glycerin. These fatty acids, or their glycerides, are capable of absorbing other elements, such as iodine or the oxygen of the air, to form saturated compounds. When oxygen is thus absorbed at a rapid rate, the oil becomes thicker as the oxidation proceeds, and eventually forms, first, a sticky layer and then a dry film. Oils which behave in this way are termed " drying oils," to distinguish them from oils in which the process takes place much more slowly (*semi-drying oils*, typified by cotton seed oil), and from those in which no appreciable change is noted for a long time (*non-drying oils*, typified by olive oil).

When linseed oil has been brought, by the action of heat and oxidation, to a stage in which it will rapidly complete the process of drying, it is known as *boiled linseed oil* or *linseed oil varnish.*

The process of oxidizing linseed oil is, for certain purposes, accelerated by the presence of " driers," which appear to act as conveyors of oxygen from the air to the hot oil. Among the substances used for this purpose are manganese borate, lead, manganese and cerium compounds of linseed oil, and compounds of certain metals, notably lead and manganese, with the acid of ordinary rosin (resinates). In the case of printing ink varnish, the driers are added to the finished product, and are suitable only for certain types of inks.

It is essential that linseed oil should be well matured before being made into printing ink varnish, for, when allowed to stand, it throws down a deposit of mucilaginous substances.

All the earlier writers from Moxon to Savage agree in regarding this period of maturing as indispensable.

Walnut (nut) oil, which was the base of varnish in some of the early French printing inks (*see* p. 84), is now mainly used as the medium for transparent oil paints, and is probably no longer used in printing inks, owing to its being expensive.

In recent years the drying oil extracted from the soya bean has been extensively used, especially in the United States, as a printing ink medium, either alone or in admixture with linseed oil. Soya bean oil has good drying properties, although it is not oxidized quite so rapidly as linseed oil, nor does it form so hard a film. Like linseed oil, it requires to be " boiled " before it is suitable for printing inks, but when thickened to the right consistence it gives excellent results in practice.

Lithographic Varnish. A different type of varnish is required for lithographic inks from that of ordinary letterpress ink. The commercial varieties of these varnishes take their name from their consistence, and are accordingly termed " extra strong," " strong," " middle," " thin," " tint," and " thin tint," those at the head of the list being the most viscous and furthest removed from the original raw linseed oil. The changes which take place during the boiling process are analogous to those already described, the oil taking up oxygen from the air, and also undergoing a change in constitution, to which the term "polymerization" is applied.

The temperatures and degrees of oxidation have to be carefully controlled throughout the boiling process, and much skill is required to produce varnishes of a uniform character.

Modern Apparatus. The old simple process of boiling the oil in a covered kettle over a free fire, and firing the vapours from time to time, has now been largely superseded by processes in which the raw oil is heated either in steam-jacketed pans or by means of a steam coil immersed in the oil. In some processes a current of air is simultaneously blown into the hot oil under pressure, whilst, in others, oxygen and ozone are brought into contact with the linseed oil.

The free-fired pan or "kettle" for making "burnt" oil by the old method is a cylindrical vessel built into a brick stove, with a surrounding space for the flames from the hearth beneath. The pan is half-filled with oil, and the flames from the vapours are extinguished by lowering a cover which is suspended from a pulley immediately over the pan.

In the more modern pans, in which the steam is admitted under pressure into a surrounding jacket, the temperature can be more closely regulated than when the pan is heated over a fire.

Various types of apparatus are used for blowing air into the oil. In some cases, cold air is forced in under pressure through holes in a pipe forming a ring at the bottom of the pan, whilst in others a current of hot air is introduced near the bottom of the vessel and the oil is meanwhile kept in motion by the action of agitator blades revolving round a central shaft.

The colour, consistence, and drying capacity of the final product vary greatly with the conditions of heating and aeration.

Thinners and Diluting Agents. The liquids used to thin linseed oil varnish or to give it certain special properties, such as a slower rate of drying, include rosin oil and rosin oil varnish, the latter being made by boiling rosin oil with lead or manganese " driers."

Paraffin and kerosene oils are also used in cheap inks to dilute the varnish. They have no drying properties, but are gradually absorbed by the paper.

Gum Varnishes. When the printing is required to have a glossy appearance, a varnish made by mixing a gum resin, such as damar or kauri resin, with hot linseed oil. Inks prepared with a medium of this kind are accordingly termed " gloss inks."

Preparation of Black Pigments. LAMPBLACK. The method of making lampblack, which has long been used in this country, is essentially the same as the Chinese method already described. In the older European apparatus, impure resinous or oily substances were burned in a vessel outside the chamber, while only a limited supply of air was admitted. The dense smoke passed into a cylindrical chamber, the walls of which were hung with sacking, and from time to time the deposited soot was scraped off by lowering the sheet iron cone suspended from the top of the chamber.

In the more modern type of apparatus, the soot from the oil or other material is caused to deposit on a chilled surface, from which it is mechanically removed by the action of scrapers.

In an apparatus of this kind the smoke from an animal or vegetable oil, or from petroleum residue, comes in contact with the hollow revolving metal cylinder, through the interior of which passes a current of cold water. The flame is thus chilled and deposits a layer of soot on the cylinder, which, as it revolves, comes in contact with the brush which removes the deposit. The cylinder is cased in by a sheet of iron casing, and beneath it is fixed the series of lamps.

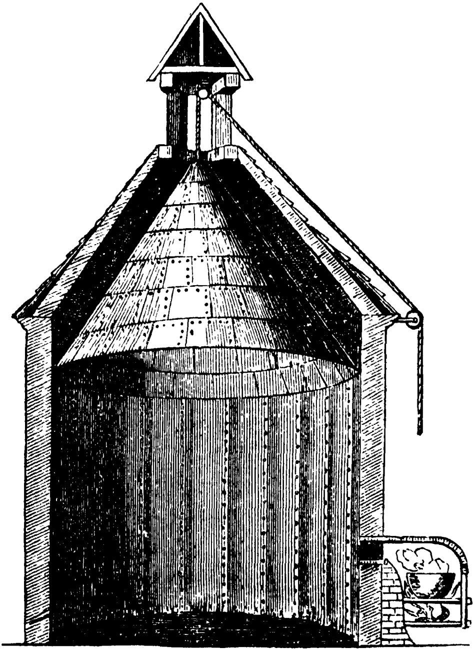


FIG. 18

OLD LAMPBLACK CHAMBER

Several much more complicated apparatus are in use, but the principle embodied in their design is essentially the same as in that described; that is to say, chilling a smoky flame by a cold metal surface and mechanically removing the deposit of soot.

Commercial lampblack varies very greatly in its depth of tone and its physical properties. Unless it has been carefully purified, it frequently has a brownish tint, due to the presence of an appreciable amount of yellowish oil deposited simultaneously with the soot.

CARBON BLACK OR GAS BLACK. Lampblack has been superseded to a large extent in printing inks by carbon black, which is now manufactured on an enormous scale from the natural gas issuing from the wells in the North American oil fields.

The method of producing it is analogous to that employed in the manufacture of lampblack, but the conditions and apparatus for obtaining the best yield of the blackest carbon have been carefully worked out.

The types of apparatus employed vary somewhat in different oil fields, but the principle of them all consists in collecting the gas in mains, and burning it in series of burners beneath moving metal surfaces, upon which the soot condenses and from which it is automatically removed by means of scrapers into bins.

The so-called " channel " system is now chiefly used. In this system the black from the natural gas is deposited upon the smooth under-surface of steel channels, beneath which, at a distance of 3 to 4 in., is a series of burners. The channels are grouped in tables of eight, and are given a slow reciprocating motion, whereby the black is scraped off them into hoppers. The quantity of the black varies with the speed of scraping, as well as with the pressure of the gas and the supply of air.

Carbon blacks have a richer tone and a greater covering and tinting power than any other kind of black. They contain from 85 to 95 per cent of carbon, 1 to 7 per cent of moisture, and small quantities of hydrogen and oxygen.

The estimated total production of carbon black in the United States in 1921 was 58,632,000 lb. The yield of black per 1,000 cub. ft. of gas ranged from 0·2 to 3·5 lb. Most of the carbon black plants are worked in conjunction with gasoline plants.

MINERAL BLACK. This is the name given to a smooth black powder, which is obtained from a carbonaceous shale. Its physical properties, notably its low absorption for oil, make it a suitable ingredient for certain types of ink.

VEGETABLE BLACK. This was originally known as *vine black*, owing to its first being made from vine stalks. It is now made by carbonizing various kinds of vegetable material, such as spent tan bark, waste yeast, or sawdust. It is used in admixture with lampblack or with bone black to give a more granular character to the ink.

BONE BLACK is made by heating bones in a closed retort, and purifying the residual carbonaceous material. When the bulk of the mineral matter is removed by treatment with acid, the product is known as " acid-washed bone black." Its black tone is inferior to that of vine black, which is therefore sometimes added to improve the colour. The physical properties of bone black make it suitable for lithographic inks, for which gas black cannot be used.

BITUMEN. The pitch-like material known as bitumen is used for various purposes, as a constituent of printing inks, as well as in the preliminary treatment of lithographic plates.

As its hue ranges from brown to black, according to the thickness of the layer in which it is spread, it is used as a constituent of some of the so-called *double-tone* *inks*. If pronounced contrast is given between the deep shadows and the lighter tints, a half-tone block reproduced in one printing with an ink of this type will give the effect usually obtained by two printings.

Coloured Pigments. Pigments of natural occurrence, like cinnabar, colcothar, ochre, and ultramarine, were first used in coloured printing inks; they were finely ground and mixed with the varnish by means of a slab and muller. Chemically-prepared pigments, like Prussian blue, were afterwards added to the colours at first available; but it was customary, even so late as 1823, for the printer to make his own coloured printing inks by hand on a small scale as required.

The use of coloured lakes made from natural pigments, such as cochineal and madder, was a still later development; whilst the discovery of the coal-tar colours made it possible to produce printing inks in a variety of hues previously unknown.

RED AND BROWN PIGMENTS. The most important mineral pigment is *vermilion*, which occurs naturally as *cinnabar* and, in chemical language, is a sulphide of mercury. The artificial product, which is much more brilliant than the natural mineral, is made by heating mercury with potassium sulphide. Apart from its cost, it has several drawbacks as a pigment for printing inks. Thus, it is liable to change colour on exposure to light and air, does not give a very uniform impression, and has a tendency to become powdery.

VENETIAN RED and INDIAN RED are the commercial names of pigments composed of clays containing iron oxide. The colour and characteristics of these pigments vary with the amount of iron present and with the mode of preparation.

BURNT UMBER is made by calcining the clay termed *raw umber*, and *burnt sienna* is prepared in a similar way from *raw sienna,* an earth which also owes its colour mainly to iron oxide.

These earth pigments are characterized by dullness of tone, and are often decidedly gritty. All the red colours in printing inks are now obtained much more satisfactorily by the use of lake pigments.

YELLOWS AND ORANGES. The natural clay pigments sold as *raw sienna, raw umber,* and *yellow ochre* vary in shade according to the proportion of iron and manganese present. They are apt to be gritty and, although fast to light, they are not much used in printing inks.

Much more valuable as pigments are the *chrome yellows and oranges,* which range in colour from bright canary yellow to deep reddish-orange, according to their mode of preparation. Chemically, they consist of chromates or basic chromates of lead; and are prepared by adding a chromate or bichromate of sodium or potassium to a solution of a lead salt, so as to precipitate a lead chromate or bichromate, the hue of which will depend upon the temperature and other conditions during the precipitation.

Although valuable pigments for printing inks, the chrome colours have the drawback of not being fast to light and of darkening, like other lead pigments, on contact with sulphur either in other pigments or in the air.

CADMIUM YELLOW is an exceedingly stable pigment but is too expensive for general use.

ORANGE MINERAL consists of a mixture of oxides of lead, and is similar to the ordinary *red lead* of commerce. It darkens on exposure to light, and is liable to form a soap with the linseed oil in the ink, which then becomes stringy, or " livery " as it is technically termed.

Blue Pigments. ULTRAMARINE is an impure silicate of aluminium which occurs naturally, but is more usually an artificial product, made by calcining a mixture of aluminium silicate, sodium carbonate, sodium sulphate, and charcoal. It has a brilliant hue, but does not make a very satisfactory printing ink, and is liable to rub off as powder from the printed surface.

PRUSSIAN BLUE, CHINESE BLUE, and BRONZE BLUE are varieties of blue pigments made by varying the conditions under which a precipitate is obtained by treating a solution of ferrous sulphate (copperas) with potassium ferrocyanide (yellow prussiate of potash) and an oxidizing agent. They are among the most valuable pigments for printing inks.

Green Pigments. CHROMIUM OXIDE is an olive green, very permanent pigment, and, as its name indicates, is an oxide of chromium. It is, however, expensive, and its dull colour limits its application in printing.

A brighter green is made by mixing chrome yellow or zinc yellow (zinc chromate) with Prussian blue in proportions varying with the shade desired. These pigments, which are sold as *chrome green*, are liable to change in colour on exposure.

White Pigments. The value of white pigments in printing inks is to dilute the colour of other pigments and so produce lighter tints. Others are used as bases to give body to the ink. The most permanent white pigment is

ZINC WHITE, which is prepared by roasting zinc or an ore of zinc, and condensing the fumes of the oxide which are formed.

WHITE LEAD, which is a basic carbonate of lead, has great covering power, but is liable to turn yellow on exposure, and has the drawback of lead pigments of incompatibility with sulphur.

LITHOPONE is the commercial name for another useful printing ink pigment. It is made by mixing together precipitated barium sulphate (*blanc fixe*) and zinc sulphide. Owing to the presence of the sulphur compound, it cannot be used.

The natural barium sulphate is known as *barytes*, to distinguish it from *blanc fixe* (the artificial pigment). It is useful to mix with pigments which take up an excessive amount of oil, but has not much covering power.

PARIS WHITE is precipitated chalk or natural chalk freed from grit. It is sometimes used as a basic substance.

ALUMINIUM HYDRATE is the commercial name for a mixture of aluminium sulphate and aluminium hydroxide, and is used, not as a tinting agent, for which purpose it is not sufficiently opaque, but to make letterpress inks work more smoothly.

Lake Pigments. In order that a vegetable colouring matter or a coal-tar dye may be in a suitable condition for mixing in printing ink, it is necessary to have it fixed upon a base such as alumina.

In some cases the colouring matter is precipitated with tannin and the precipitate ground up with the basic material ; in others, it is precipitated direct on to the base.

In the case of certain lakes, it is necessary to add a mordant, such as tartar emetic, to prevent the colour from separating afterwards.

Some of the earlier lakes, such as carmine lake, derived from cochineal, are fugitive colours, and this also applies to many of the aniline dye lakes ; but madder lakes are stable colours, and most of the aniline lakes made from diazo dyestuffs will resist the action of air and light better than most mineral pigments.

The use of pigments of this kind is essential for printing inks for three-colour work, for it would not be possible to obtain the correct colours from the limited choice of the older type of pigments.

Mixing the Pigment and Varnish. The quality of a printing ink depends not only on the choice of suitable pigments and varnish, but also upon the thorough incorporation of all the ingredients into an absolutely uniform mass.

In the case of black inks, the proportions of the various blacks, oil varnish, soap, thinners, etc., will depend largely upon the kind of work for which the ink is intended. For example, the proportion of black in the published formulae for certain German inks ranges from about 19 to 40 per cent, varying according to whether the ink is to be used for rapid newspaper work, or for printing books or illustrations.

The weighed quantity of varnish is put into a mixing machine ; the weighed amounts of pigment, sliced soap, driers, and other chosen ingredients then added ; and the whole incorporated into a uniform mass by the blades of the machine.

A small machine, which can be worked either by hand or by power, is shown in Fig. 19. This takes a charge of 5 galls., which is effectively mixed by the rotation of the vertical blades, which are brought into the pan by raising the lever above the flywheel.

A larger machine, the action of which is based upon the same principle, is represented in Fig. 20. In this case the mixing is effected by two sets of vertical blades, which are driven by means of cog-wheels.

After the pigment has been thoroughly incorporated with the medium, the *pulp*, as it is now termed, is transferred to a grinding mill and reduced to a fine homogeneous paste free from any lumps. These mills, a typical example of which is shown in Fig. 21, contain a series (often 6 or 9) of rollers of hard, fine-grained stone, such as porphyry or, in some machines, of chilled steel.

The ink is removed from the surface of the rollers by means of a scraper or " doctor " attached to the roller bearings. In the case of poorer qualities of ink, sufficient grinding can be effected in a mill of the type shown in Fig. 22. The trough varies in diameter from 1 to 8 ft., and the grinding is done by the edge of a steel roller. The ink is continually removed from the roller and the interior of the pan by means of scrapers of a special form.

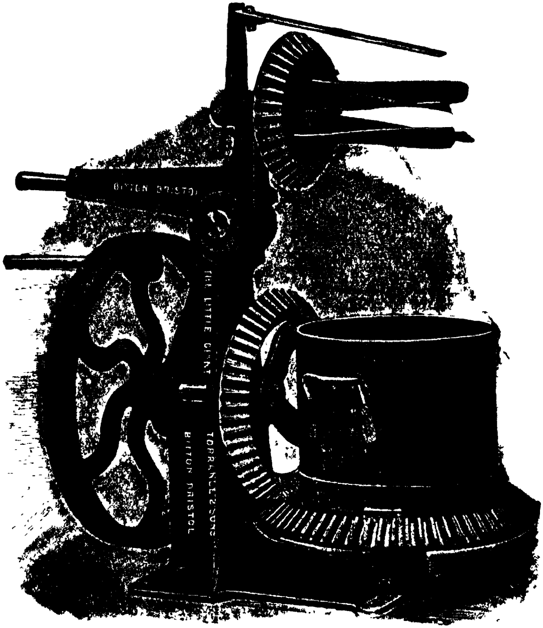


FIG. 19

TORRANCE'S SMALL MIXING MACHINE

Inks for Three-colour Printing. The application of photographic methods to the reproduction of coloured objects by means of process blocks has led to the manufacture of printing inks, the colours of which are selected in accordance with the scientific principle upon which the invention is based.

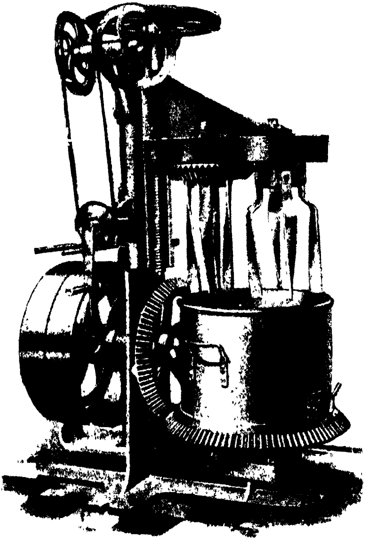
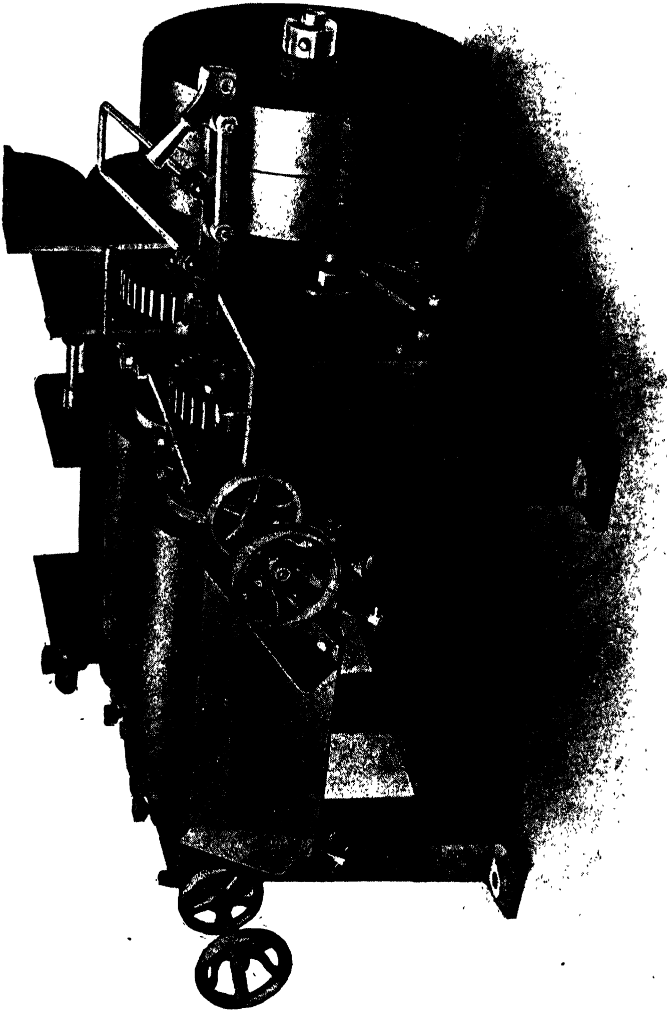


Fig. 20

TORRANCE'S "PATENT" MIXER

It is common knowledge that a photograph taken in the usual way upon an ordinary plate does not correspond in its lights and shades with the colours of natural objects, but represents violet and blue colours as white, and greens, yellows, and reds as hues approaching to black; whereas, in the case of direct daylight, the most



intense light is to be seen in the yellow portion of the spectrum, whilst the red and violet ends are both dark.

Colour Sensitive Plates and Screens. This falsification of colour by photography was first corrected by Vogel's discovery that it was possible to render the sensitized gelatin film of a photographic plate much more sensitive to the light reflected from coloured objects by adding certain aniline dyes to the emulsion used for coating the plate.

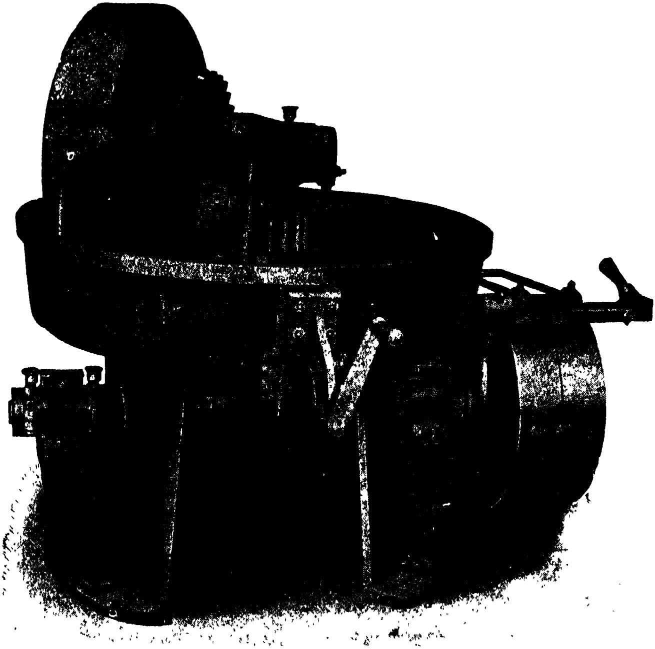


FIG. 22

THE TORRANCE MILL

Photographic plates thus made sensitive to colour are termed " isochromatic " or " orthochromatic " plates, and are used in association with an appropriate colour screen or light filter fixed in front of the camera lens.

The principle upon which these screens are made for three-colour work was discovered by Clerk-Maxwell in 1861, long before the invention of isochromatic plates. These curves were the outcome of a series of experiments which proved that by taking three photographs of the same object, each through a screen of a different colour, and then blending the three pictures upon a screen with the aid of lights of a suitable colour, it was possible to obtain an approximately true reproduction of the original colours.

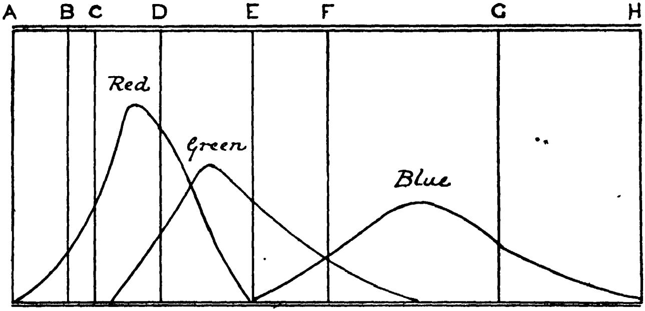


FIG. 23

CLERK-MAXWELL'S COLOUR CURVES

The proportions of the spectrum to be included in the three colour screens are shown diagrammatically in Clerk-Maxwell's curves (Fig. 23). From these it will be seen that the red screen must include a proportion of yellow and green ; the green screen a proportion of orange, yellow, and blue ; and the blue screen a large proportion of violet and a small proportion of green.

In practice, screens can be made in colours approximating those corresponding with the curves by staining gelatin films on glass plates with suitable mixtures of aniline dyes. The three negatives of the coloured object are made on colour-sensitive plates, and each with its appropriate screen, and these negatives are used for the preparation of three half-tone blocks in the usual way. The printer has then to make three successive imprints from these blocks with inks of such colours that the final blended result shows an approximate representation of the colours of the original. The more closely the colours of the inks agree with those of the colour screens, the truer will be the reproduction of the coloured object.

Pigments for Three-colour Work. From what has been said, it is obvious that the pigments chosen by the maker of the printing inks should coincide with those used by the makers of the colour screens, which is not always the case. The printer has to depend upon the use of three pigments—blue, red, and yellow—of as pure a hue as possible, and to obtain the intermediate colours, corresponding with the overlapping portions of the colour curves, by the superposing of the three.

Thus, in the darkest portions of the reproduction, the dots of the half-tone block of the three foundation colours are superposed, whilst, in other portions, dots of one colour are printed over one another so that they are blended by the eye into a mixed colour.

This explains why it is necessary to use transparent inks, so as to obtain the various blends of colour. As the bright yellow pigments commonly used in this kind of work are not transparent, they are used for the foundation or first printing, whilst the red and blue blocks are printed with transparent pigments.

Lakes made from alizarine or aniline dyes are used for the red pigments, and Prussian blue is commonly used to obtain the blue tints, although lakes of various organic dyes are now made in various colours, and can be used as the basis of printing inks which are both transparent and permanent.

It is necessary that the inks of the three colours should have closely similar covering power, in view of the fact that they have to be blended in various proportions to reproduce the colour gradations of the original object.

A considerable amount of work has been done on the subject of standardizing the colours both for the inks and for the screens, and Professor Ostwald has recently devised a colour scheme whereby it is possible to specify and obtain a pigment in any required shade of any colour. The adoption of this scheme by the makers of colour screens and of printing inks would greatly simplify the task of the printer who undertakes three-colour work.

Rotagravure Inks. The rotagravure process, which came into general use in this country about eight years ago, takes its name from the fact that it enables both illustrations and text to be printed simultaneously on rotary machines working at a high speed.

The essential principle of the process is the opposite of that used in the half-tone process, since the printing surface, instead of being made up of a series of minute dots differently spaced, is an intaglio composed of minute rectangular cells, approximating 40,000 to the square inch, and varying considerably in depth. The effects of light and shade are obtained by the variations in the thickness of the film of ink transferred from the cells to the paper, a very wide range of tones being thus obtained.

In the process of printing, the surplus ink is removed from the cells, walls, and surrounding margins by means of a fixed "doctor" blade, and the graduated film of ink left in the cells is brought into contact with a web of paper by means of an impression roller, which gives the necessary pressure to lift the ink from the cells on the cylinder.

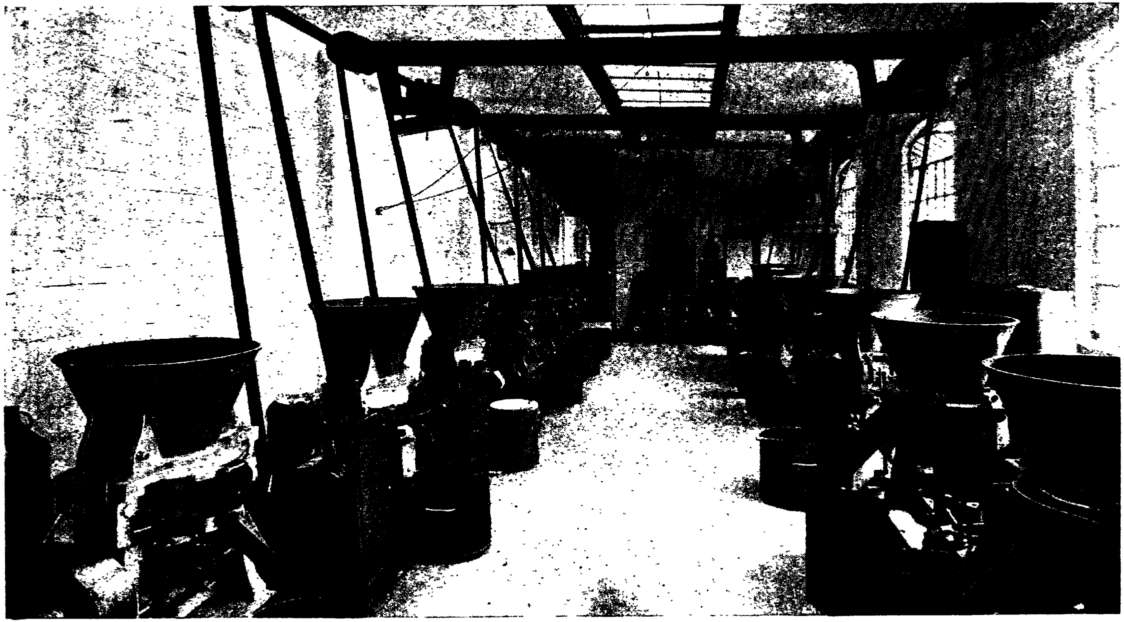
The main essentials required by the ink for this process are that it shall flow freely, be capable of being lifted completely from the cells, have a greater affinity for the paper than for the cylinder, and dry rapidly. In addition, it must not form precipitates of pigment in the cells during the printing ; must be transparent without being deficient in density ; and, above all, must be particularly finely ground.

To secure these combinations of qualities in an ink was a difficult problem, and it was only after long experiments with different mixtures of pigments with volatile and non-volatile media that inks fulfilling all these requirements have been prepared. ✓

The ordinary ink-grinding mills were soon found to be unsuitable for rotagravure inks, since they allowed the volatile spirit media to evaporate too rapidly, and cone-shaped mills were therefore introduced.

The general form and arrangement of these is shown in the illustration (Fig. 24), which represents two lines of the machines in the department reserved for the manufacture of these kinds of ink in the works of Messrs. B. Winstone & Sons, to whom the writer is also indebted for the details of this new development in the history of printing and printing inks.

Each of these mills contains two horizontal superposed steel discs, which revolve and grind the mixture of pigments and media into an absolutely fine and uniform mass, which finally escapes through channels cut in the opposing faces of the discs.



By the courtesy of *Messrs. B. Winstone & Sons, Ltd.*

FIG. 24.—A ROTAGRAVURE INK FACTORY

Showing special mills for grinding rotagravure inks

Printing Inks for Bank Notes and Cheques. The ease with which ordinary iron-gall ink can be removed from paper without leaving any very apparent residue has led to the adoption by bankers of various printed devices, which would simultaneously be affected by the reagents used to remove the writing ink, and so reveal any attempt at fraud.

Savage, in his book *The Preparation of Printing Ink* (1832), gives an account of an ink of this type which was kept secret for many years " by one house in the City of London." This black ink, which was used for the printing on cheques and the like, was made from an infusion of galls, iron, and logwood. The mixture yielded a black deposit which was separated, dried, and ground up with soap, turpentine, and copaiba balsam. Characters printed with this ink would be bleached on contact with an acid, alkali, or bleaching agent.

Savage also recommended that, as an additional safeguard, the face of the cheque should be printed over with a fine lace pattern in a light-coloured ink, which would be removed by the action of an acid. He suggested for this ink, ordinary carmine lake ground up with printing varnish and curd soap.

Various devices of this nature have since been adopted, in addition to the use of " safety inks," for the written portion of the cheque, and many interesting details on the subject can be gathered from a study of the various patents of inks claimed for the purpose.

A process to prevent photographic reproduction was patented by Scropyan in 1857, the body of the cheque being printed in yellow, red, or orange, whilst the design was printed in another colour which " absorbed light." An ink used for the purpose was composed of a mixture of iron hydroxide, gallic acid, and boiled linseed oil. This, of course, was prior to the invention of isochromatic plates.

Another early process, patented in 1859, consisted in incorporating the paper pulp with a pigment such as chromium oxide, whilst the printing ink contained china clay and sulphur.

An adaptation of an old idea was suggested by Pinkney in 1876, the paper of the cheque being impregnated with a substance, such as a ferrocyanide, which would react with ordinary writing ink to form an insoluble compound (Prussian blue). Another simple plan was that patented, in 1879, by Nesbit, who incorporated an aniline dye, soluble in water, with the lithographic ink, so that any attempt to remove writing from the cheque with an aqueous agent would cause a smudge.

A much more effective process is that devised by Hehner and Dupré in 1881. An ink suitable for printing invisible designs on the cheque is made from a mixture of a sulphide of a heavy metal, such as zinc, with a salt of another heavy metal, such as lead carbonate, with a suitable medium of dextrin, gum, or the like. If characters printed with this preparation come into contact with an acid, alkali or cyanide, a dark stain is at once formed on the paper, owing to the interaction of the ingredients in the ink.

Mention may also be made of another method requiring the paper pulp to be impregnated with a chemical. In this process, patented by Rowan in 1904, the pulp is mixed with potassium ferrocyanide and ammonia, and the designs are printed with an ink to which ferric chloride, potassium iodide, and caustic soda have been added. The oil of the printing ink prevents these various ingredients from acting upon each other ; but if an acid or alkali is applied, they are brought into solution, and will then react and produce stains on the paper.

Methods of Examining Printing Inks. Chemical analysis is useful sometimes to ascertain the presence of certain pigments or constituents of varnish in printing inks, but a full analysis of the complicated mixtures now produced is rarely possible ; nor would it convey much serviceable information to the printer, who requires the ink for a particular purpose and, provided it gives the desired results, regards its composition as matter of second importance.

The maker of the ink, however, requires the aid of chemical examination in the selection of his raw materials. For instance, it has been shown that there is a relationship between the behaviour of carbon blacks in printing ink and their chemical composition, which may vary in the proportion of carbon between 85 and 90 per cent.

The tests of the physical characteristics of mineral pigments is of even greater importance than the chemical examination. One of the principal points to which attention is directed is the colouring power or "strength." This is determined by mixing a weighed quantity of a pigment, such as carbon black, with measured quantities of oil and pure zinc oxide, and noting the tint produced, in comparison with that given by a standard black (or coloured pigment) under parallel conditions.

The value of a pigment also depends upon its covering power, which, in the case of lampblack, may be estimated by mixing weighed quantities of the pigment and of oil and spreading the mixture over paper until the surface below becomes visible. Then a measurement of the area thus covered, in comparison with that covered by a standard pigment, will give the relative covering powers of the two pigments.

Pigments also differ very greatly in their behaviour when mixed with oil. Some are what are technically known as " short " pigments ; that is to say, they yield a stiff mixture, even when incorporated with a very large amount of oil. Others " flow " well or, in other words, run smoothly, and can be distributed easily over the rollers of the press. At the same time, the ink, while flowing well, must have " length " (i.e. the capacity to be drawn unto strings), this being dependent upon the " tack " or degree of adhesion between the particles.

Certain pigments are incompatible with others. For example, lead pigments cannot be used with any pigments containing sulphur, since lead sulphide will be formed and the ink will turn brown. Lead pigments also combine sometimes with the linseed oil to form a lead soap which is left distributed throughout the mass. This trouble is known as " livering," from the liver-like appearance of the soap.

Other pigments are liable to " bleed " ; that is to say, they are partly soluble in water or in oil, and so, instead of remaining as a fine powder distributed through the oily medium of the ink, they are partly dissolved, and the oil may then separate on the paper and cause some of the dissolved pigment to run. This fault is most liable to occur with some of the aniline dye lakes, and these are accordingly unsuitable for use in printing inks.

Stress has already been laid upon the importance of having the pigment free from gritty matter and upon its uniform incorporation with the oil medium of the ink, and these are among the principal points to which attention is directed in the examination of the finished ink.

In addition to tests on all these points, an examination of a printing ink should also include tests as to the presence of the colour to light and air, the rate of drying for the particular purpose required, and the behaviour of the ink upon the paper that is to be used. In short, as in the case of marking inks, physical tests and trials under conditions similar to those of the actual printing process will be found of more value than chemical examination, though the latter may often be required to explain the cause of observed defects in an ink.

IX

TYPEWRITING INKS AND CARBON PAPER

TYPEWRITING INKS

The invention of the typewriter and its general adoption have led to the manufacture of a new type of ink, which, in some respects, may be regarded as intermediate between writing and printing inks. In most of the typewriters now in use, the ink is applied to the face of the movable type either by means of a stretched ribbon or of a pad saturated with the ink, and is then transferred to the paper by the further action of the levers which control the type.

The earliest kinds of typing inks consisted of solutions of aniline dyes, such as methyl violet, with a small amount of a thickening agent, such as glycerin. Inks of this type, which are still to be met with, have the drawback of giving writing which is liable to smudge. The glycerin in the ink also causes the ribbons to absorb moisture from the air, thus altering the composition of the ink. This defect was remedied by using hot oleic acid (the chief fatty acid in vegetable oils) as the solvent, and diluting the ink to the required consistence with a mineral oil. Nigrosine was used as the dye in black inks, whilst various basic aniline dyes were used in coloured inks.

Modern Inks for Typewriters. In the more recent processes, the use of soluble dyes has, to a large extent, been superseded by that of insoluble pigments similar to those used in printing inks. As in the case of the latter, it is essential that mineral pigments should be ground extremely fine, since any grit will have an injurious effect upon the metal of the type. Cylinder mills, similar to those used in preparing artists' colours, are used for this purpose, and the grinding is repeated several times until the material is absolutely uniform.

The pigments used for black ink are lampblack and carbon black, whilst the mineral coloured pigments include Prussian blue for blue ink and antimony cinnabar (made by heating antimony sulphide in a current of air and steam) for red inks. Vermilion is too expensive for this purpose. Lakes prepared from aniline dyes in the same way as described under printing inks are also common pigments in typing inks. Where necessary, the hue of the pigment is rendered lighter by a suitable addition of zinc white, and special shades are obtained by blending various pigments. In short, the methods used in preparing printing inks are more or less applicable to inks for typewriters.

Mixing the Inks. The finely ground pigment or mixture of pigments is first ground to a smooth paste with vaseline or a thick mineral oil to which a small addition of paraffin wax is sometimes made. The mass is then incorporated with about 10 per cent of a solution or mixture of a suitable pigment in oleic acid, or a fatty oil, and the grinding then continued until the whole ink is perfectly uniform.

A typical black ink is made by grinding together 10 parts of pure lampblack with 40 parts of vaseline, and then adding 5 parts of a 50 per cent solution of nigrosine in oleic acid. Ink of this kind gives black impressions which cannot be removed from the paper without showing indications of the process.

Like the coloured inks made from mineral pigments, this black ink is not suitable for copying purposes.

Copying Inks for Typewriters. When a copy of an impression is to be taken in a copying book, the ink must contain a pigment which is soluble in water. The aniline dyes, which are or have been used for the purpose, include methyl violet, methylene blue, malachite blue, and safranine. They are ground up with vaseline or, in some cases, with a fatty oil, such as castor or fish oil. A German formula for the preparation of a hectographic ink capable of yielding fifty or more copies, consists in dissolving 100 parts of crystal violet in 300 parts of glycerine and 190 parts of water and then adding 60 parts of dilute hydrochloric acid (to increase the solubility of the dye) and 50 parts of a 50 per cent solution of dextrin. It is claimed that ribbons impregnated with this ink lose a considerable portion of the free acid by evaporation ; but as the ink still remains decidedly acid, it is necessary to wash the type with water after using the ribbon.

Ribbons for Typewriters. Certain properties are required in the fabric of the ribbons which are to con vey the ink to the face of the type. They must be strong and yet flexible, and the edges must be so woven as not to be displaced when struck by the type. The earliest ribbons were made of silk, but at the present time they consist of a cotton fabric with a very fine mesh specially woven for the purpose.

In the early days of their manufacture, all ribbons were saturated with the ink by passing them under rollers at each end of the ink-bath, and then through rollers in another vessel to remove the excess of ink. This process, which is still used in some works, produces ribbons which give very clear impressions, but which are soon exhausted of their ink—long before the fabric of the ribbon itself is worn out.

This drawback is overcome in more recent processes by attaching a thin layer of the ink to each side of the ribbon. For this purpose the ribbon is made to pass in a continuous length between rollers, one of which is coated with a film of the pasty ink, which is transferred to one surface of the ribbon by the pressure of the second roller. The other side of the ribbon then receives its layer of ink in the same way, and finally the finished ribbon is wound on to spools and cut into the measured lengths by means of a mechanical device. In order to prevent evaporation of the ink, the ribbons are packed in tin boxes with closely-fitting lids.

CARBON PAPERS

Although typing inks which will yield a copy when pressed with moistened paper are still used to a limited extent, they are steadily being displaced by carbon papers, which enable several copies to be taken at the same time as the original typing is done. The principle used in the manufacture of these carbons is that of impregnating or, rather, coating one side of a suitable paper with a pigment mixed with a basis of oil or wax in such proportions that, when placed face downwards over white paper and beneath the sheet upon which writing or typing is being done, the pigment is transferred by the pressure of the pencil, pen, or type to the lower sheet, and an exact copy of the writing obtained.

The nature of the pigments and media used varies with the purpose for which the paper is required, and the various formulae employed by different manufacturers are naturally guarded as trade secrets. There are, however, numerous details which are common property to all engaged in the industry, and the writer wishes to thank Mr. C. T. Smoothy for many of the following particulars of the preparation of various kinds of carbon papers.

One of the earliest published methods is that claimed by Carter in an English patent of 1903, according to which the backing for the sheets consists of a mixture of oil, glycerin, and printing ink. The presence of too much glycerin or of free fatty acids in the oil is, however, objectionable, and not more than a trace of either should be detectable in a good carbon paper.

Paper for Carbon Papers. The character of the paper used as the basis is a matter of great importance, if uniform products are to be obtained. For pencil carbons, a paper of rag and pulp is suitable, but for pen carbon papers only rag pulp is used, and the paper is bought in accordance with the results of chemical analysis and mechanical tests. In the case of carbons for typewriters, the paper must be of such a texture as to receive the blow of the type uniformly and yield clear imprints without needing the application of undue force.

Pigments for Carbon Papers. The old carbon papers used in manifold books for copying pencil writing were coated, frequently on both sides, with a composition of lampblack, oils, and waxes. Lampblack and carbon black are still used as the black pigment for different kinds of carbon papers, whence the name of " carbons," which is retained even when the paper is coated with a coloured pigment. The richer tone of carbon or gas black makes it a more suitable pigment than lampblack for this purpose.

Prussian blue is used as the pigment for some of these papers, but has the drawback of requiring excessive care in grinding, so as to obtain a product free from grit. The other colours used are blue, purple, green, and red lakes of a similar type to those mentioned in the Section on " Printing Inks."

The pigments are ground up into an ink with a suitable proportion of wax and oil. The waxes used for the purpose include residues of carnauba wax, montan wax, and ozokerite and paraffin waxes; whilst mineral oil, petroleum jelly (vaseline), and oleine form the thinning agents.

The incorporation of the pigment with the oil and wax medium is effected by grinding the weighed quantities of the ingredients between rollers of granite and steel until the paste is perfectly smooth and uniform. This part of the process is closely comparable with the grinding of printing inks previously described, and requires just as much care. The final product is a black or coloured ink which differs, however, from printing ink in not containing a drying oil or varnish.

Coating the Paper. The paper is supplied by the mills in rolls 18 and 26 in. wide, and averaging from 70 to 80 lb. in weight. The roll is connected with the coating machine and made to pass over rollers which can either be heated by steam or chilled by water internally, as required; and a fan or other device for rapidly cooling the surface of the rollers is also provided.

The rollers, as they turn, bring the surface of the paper into contact with the ink in the ink pan, and the amount of ink thereby applied to the paper is regulated by means of "equalizers," so that just sufficient ink to congeal readily, without leaving a sticky surface, is delivered.

The success of coating the paper by this method depends largely upon the skilled judgment of the workman.

When the coating of its face is complete, the roll is wound again upon a drum of known diameter, so that it can be cut into special sizes, such as demy, double crown sheets, etc.

In manufacturing certain pen carbon papers, a brush, termed a "jigger," is affixed to the machine, and by its vibratory motion gives a wave-like appearance to the paper. This is usually seen in papers made in weights below 7 lb., and the process is also applied to some typing carbons.

Some papers are also made with selvedges, so that, if required, they can be gummed into manifolding books.

The trade numbers of pencil papers (7, 10, 15, 18, and 20) represent the weights of the paper in pounds, prior to coating of 500 sheets, each 20 by 30 in.

In the case of pen carbon papers, the weights are 5 lb., 7 lb., and sometimes, for heavy work, 10 lb.

Pencil carbons are coated on either one or both sides of the sheet, and are accordingly termed " semi " or " full " in the trade.

X

INKS FOR VARIOUS SPECIAL PURPOSES

SAFETY INKS

It has been shown in the section on the characteristics of ink in writing, that ordinary iron-gall inks can be removed by treatment with bleaching reagents, leaving little, if any, trace of the original writing. On the other hand, the lampblack or other form of carbon in a carbon ink resists the action of chemical agents, and the pigments in the old inks were, therefore, more stable than those of modern inks.

Various devices for printing bank notes or cheques with special forms of ink that would be altered in colour by any attempt to remove the writing from the document have been described (p. 106), and attempts have also been made, from time to time, to safeguard the writing itself by the use of an ink that would not be affected by the chemical agents which bleach ordinary writing ink.

A comparison of the behaviour of carbon inks and of iron-gall inks in this respect naturally suggested the idea that by writing with a mixture of the two inks the advantages of both would be obtained. This suggestion has frequently been put forward in various forms, but is rarely satisfactory, partly for the reason that the two kinds of ink do not mix well, and partly owing to the fact that it is usually possible to remove the carbon particles mechanically by brushing the surface of the paper while immersed in a reagent.

What is required, therefore, in a " safety " ink is that the carbon pigment should be in such a form that it will not merely be attached to the surface of the paper, but will also penetrate, like ordinary writing ink, into the fibres, and so resist mechanical attempts to remove it without simultaneous perceptible injury to the paper itself.

The invention of such an ink was regarded of such importance in the early part of last century, that, in 1831, a Chemical Commission was appointed in Paris to investigate the question. Two inks were recommended in the Report of this Commission as fulfilling the required conditions. The first of these was a mixture of dilute hydrochloric acid with about 0·5 per cent of Indian ink ; whilst the other was composed of a solution of manganese acetate acidified with acetic acid and mixed with Indian ink. In this case, the writing had to be exposed to the fumes of ammonia to make it indelible.

Experiments made by the writer have shown that the second formula gives the better results, but that the final writing is not a good black. It is also open to the objection that it requires final chemical treatment to fix the writing.

Various preparations of lampblack incorporated with different media, to keep the carbon in suspension, were then proposed, such as the alkaline shellac ink of Desmarest (1831), and the ink patented by Stephens and Nash in 1837.

An ink which came into practical use was that made by Traill in 1840. This consisted of a mixture of lampblack and indigo with a glutinous medium prepared from wheat flour. It was adopted by the National Bank of Scotland as a means of preventing forgery, but has long been discarded, probably owing to its being a less effective safeguard against fraud than the use of cheques printed with special inks (p. 106).

In another early ink of this type, a solution of sodium silicate (water-glass) was incorporated with lampblack. Ink prepared in this way gives very black writing, which resists the action of chemicals, but does not penetrate well into the paper, and much of it can be afterwards removed by brushing the surface of the paper under water.

Use of Colloidal Carbon. The best of these early preparations were in all probability those made from Indian ink of such quality that the carbon in it was in a colloidal state, that is to say in a form intermediate between a solid and a liquid condition. Lampblack can be obtained in a state more or less approaching this condition by a careful fractionation of the smoke into heavier and lighter grades, and the commercial Chinese ink shows great variations in its properties according to the grade of lampblack from which it was prepared. This explains why the safety ink of the Paris Commission had good penetrating qualities, whereas some of the other "security" inks, in which crude lampblack was used as the basic pigment, gave writing which could be washed off the paper.

The best known form of colloidal carbon on the market is Acheson graphite, which is made artificially, and is then treated with tannin in such a way as to yield a product in so fine a state of division that it will remain suspended in water for a very long period, unlike ordinary lampblack, which, if mixed with water, will quickly form a solid deposit. Colloidal graphite particles will penetrate the fibres of paper readily, and it has therefore been made the basis of several "anti-fraud" inks. The chief objection to its use is that, compared with carbon black, it is of a poor hue. Hence, other forms of carbon are mixed with it, and indigo and aniline black or other dyes are dissolved in the liquid. These give colour to the writing, and make it resemble that given by an ordinary black ink, but the "safety" factor of the ink is the colloidal carbon.

Safety Inks with Special Papers. Various methods have been suggested for impregnating the paper with one chemical, and writing upon it with a solution of a second chemical which would combine to form the ink in the process of writing. For example, by impregnating a paper with potassium ferrocyanide and writing upon it with a solution of an iron salt, the characters appear in Prussian blue.

Devices of this kind might offer some protection against an unskilled forger, but would not be effective against treatment with suitable chemical reagents. The records of the Patent Office show that many devices of this kind have been patented, the earliest instance being the patent of Stevenson (1837), according to which the paper was saturated with manganese chloride and potassium ferrocyanide. It is doubtful, however, whether any of these inventions met with any material commercial success, and in any case the adoption of special inks for printing cheques must have made the process of little value.

INK POWDERS AND TABLETS

It is interesting to note that the first English patent on the subject of ink, that of Holman in 1688, claimed the use of a powder which, when mixed with water or beer, would form a black writing ink. Unfortunately, as is the case with all the earliest patents, no particulars of the process are given in the specification.

Another early patent, of which also no details are published, is a process of making ink into a cake or solid, which was claimed in 1768 by Dring. In the old type of iron-gall inks, in which the oxidation was allowed to proceed so far that the final product was a partially insoluble iron tannate kept in suspension by means of the added gum, a sort of solid ink could have been made by evaporating the liquid to dryness. The residual powder would then only have required the addition of water to reproduce an ink, although a much poorer one than the original preparation ; whilst if the oxidation of the tannate was complete, the new ink would have had more of the character of a varnish than of a writing ink.

It is the change in an iron-gall ink from a soluble to an insoluble tannate which must always interfere with the preparation of an iron-gall ink of the modern type by any method of this kind.

Another way of approaching the problem is to mix the dry ingredients together in the right proportions to form the ink on the addition of water. A preparation on these lines consisting of powdered galls, copperas, and gum was described by Canneparius in 1660, and the idea was included in an English patent in 1867. It is obvious from what has been said in the preceding pages that the ink made from such a powder would contain a large deposit, and that ink made from it would not be ready for use until some time after the water has been added.

What is required in an ink powder is a preparation which can be dissolved in water, without leaving any residue, and which will give an ink ready for use immediately after mixing.

Several formulae for logwood inks of this kind have been published, the logwood ingredient being in the form of logwood extract or of its active colouring agent, haematein. There have also been several patents taken out for the preparation of ink powder from aniline dyes, mixed in various proportions with a thickening agent such as sugar.

It is hardly practicable to make a standard iron-gall ink in the form of a powder, since, even if pure tannin is used for the purpose, the ink will require to mature for a certain time.

The use of gallic acid to make an " acid-free " ink of the type already described gives better results for ink powders, and a suitable mixture of that acid with powdered ferrous sulphate and an aniline blue dye will dissolve rapidly, and give an ink which can be used at once.

Ink powders prepared by some such method as this are suitable for compression into tablets, and these are to be obtained in standard sizes, so that by placing the specified number in a fountain pen and filling the reservoir with water the pen will be ready for use again after a few minutes.

STAMPING INKS

The inks required for saturating the pads used with metal or rubber stamps require certain special characteristics. They must be thick enough to give a sharp impression on the paper without running or smudging, must not dry up in the pad, but yet must dry rapidly after being applied to the paper.

These opposite qualities are obtained by balancing the various ingredients. For instance, the addition of glycerin prevents the ink from evaporating in the pad ; whilst rapid drying is effected by adding a suitable proportion of methylated spirit, acetone, or other volatile solvent.

For example, a violet ink suitable for rubber stamps is made by mixing 3 parts of methyl violet, 15 parts of alcohol, and 15 parts of glycerin.

For more indelible stamping, such as cancelling documents, an ink more on the lines of printing ink is required. In the published formulae for a cancellation ink to be used with rubber stamps, a mixture of linseed oil varnish, lampblack, and finely powdered iron chloride is given.

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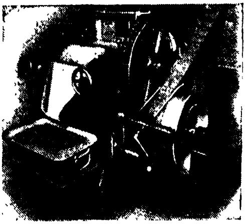

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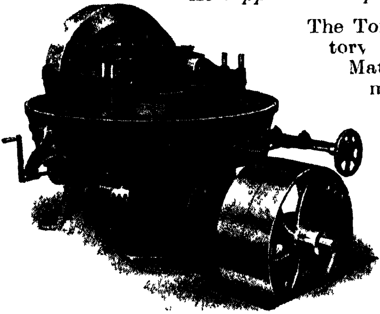

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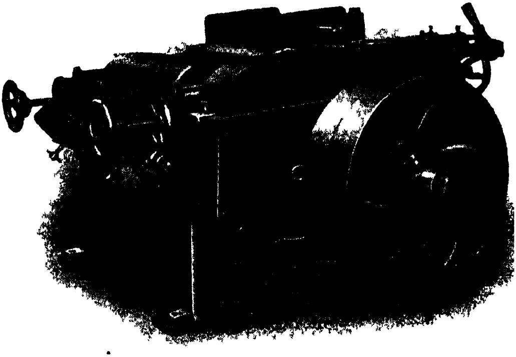

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